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THE DEVELOPMENT OF RAPIDLY SOLIDIFIED
STAINLESS STEEL ALLOYS FOR POWDER
METALLURGY APPLICATIONS - PHASE I

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STAINLESS STEEL ALLOYS FOR POWDER
METALLURGY APPLICATIONS - PHASE I

Final Report
by:

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Les principaux objectifs de ce projet étaient les suivants: modifier un appareil de coulée rotatif dans le but d'obtenir des rubans d'acier inoxydable SR (par solidification rapide) et déterminer leur résistance à la corrosion. Ces objectifs furent atteints et ce procédé a amélioré la résistance à la corrosion de ces aciers.

La coulée d'acier inoxydable par ce procédé est rendue difficile par la nature corrosive des échantillons liquides ferreux. Les composantes en céramique requises dans certaines parties de l'appareil rotatif se sont avérées très fragiles. On a toutefois réussi la coulée de plusieurs aciers inoxydables par ce procédé malgré les problèmes rencontrés.

L'examen de ces rubans a indiqué que leur microstructure était microcristalline. D'après les essais de corrosion auxquels furent soumis tous les rubans, la résistance à la corrosion locale de tous les échantillons SR fabriqués à partir d'alliages standard des séries 300 et 400 s'est avérée supérieure à celle des échantillons corroyés. Un échantillon SR d'un acier du type 304, mais dont la teneur en chrome avait été réduite, a fait preuve d'une résistance à la corrosion locale égale à celle du même acier corroyé.

Des essais de traitements thermiques ont démontré que les alliages SR pouvaient être soumis à des températures allant jusqu'à 1000°C pendant une heure sans subir une diminution importante de leurs propriétés initiales. Cette dernière qualité peut être utile dans les procédés de fabrication qui requièrent du frittage.

Plusieurs poudres commerciales du type 316L produites par atomisation à l'aide de jets d'eau ont été examinées et se sont avérées microcristallines. Ces poudres pourraient être une source commerciale de matériau SR même si leur niveau de contamination par l'oxygène est élevé.

Nous recommandons de poursuivre les travaux sur les aciers inoxydables solidifiés rapidement (SR). Ces recommandations portent sur le développement d'alliages différentes, l'étude de leur solidification et la production de poudres atomisées exemptes de contamination par l'oxygène.

FINAL REPORT

THE DEVELOPMENT OF RAPIDLY SOLIDIFIED
STAINLESS STEEL ALLOYS FOR P/M APPLICATIONS

for

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26TH MARCH, 1986.

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SUMMARY

The goals of this project included the modification of a melt spinning apparatus to produce strips of RS stainless steel, and the characterization of the corrosion resistance of RS stainless steel strips. These goals were successfully achieved, and the corrosion properties of the RS stainless steels were improved by this form of processing.

The melt spinning of stainless steel is made difficult by the corrosive nature of the molten ferrous sample. Ceramic components were required in key areas of the melt spinning apparatus, and these were found to be susceptible to breakage. In spite of the problems encountered, melt spun strips of a number of stainless steel alloys were produced.

An examination of the microstructures of the melt spun strips verified that they were microcrystalline. Corrosion tests were carried out on all strips. The localized corrosion resistance of all RS samples made from standard 300 and 400 series alloys was greater than that of the wrought samples. A RS sample of a modified 304 alloy with a reduced chromium level was found to have localized corrosion properties as good as wrought 304.

Heat treatment tests demonstrated that the RS materials were able to withstand temperatures of up to 1000°C for an hour without significant degradation of the RS properties. This may be important for manufacturing processes requiring sintering at some stage of the process.

A number of commercially available water atomized 316L stainless steel powders were examined and were found to be microcrystalline. These powders could present a commercial source of RS material, although water atomized powders tend to contain high levels of oxygen contamination.

Recommendations are made to continue the investigation into RS stainless steel products. These include development of alternate alloys, study of consolidation, and the production of low oxygen content atomized powders which are free of oxygen contamination.

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1. INTRODUCTION

Stainless steels are used extensively in many areas of industry, construction, food services and medicine. The most important property of stainless steels is their high corrosion resistance. This is a result of their high nickel and chromium contents.

It would be desirable to improve the stainless steel alloys by increasing their corrosion resistance. To be more cost effective, and to reduce the dependence on strategic materials, it would also be desirable to reduce the chromium and nickel content of stainless steels, without sacrificing their corrosion properties. Some attempts to do this have used the addition of silicon and aluminum to ferrous alloys to produce corrosion resistant materials [1]. These alloys tend to have poor mechanical properties and are more difficult to process. Another approach is through the use of Rapid Solidification (RS) processing.

In an RS process, a molten alloy is cooled to a solid state at a rate high enough to form a non-equilibrium structure. RS alloys can be characterized by a reduced grain size, a high degree of chemical homogeneity, the possibility of non-equilibrium solute concentrations, and enhanced mechanical and corrosion resistance properties.

Hashimoto et al found that an amorphous Fe-Cr-13F-7C alloy was much more resistant to corrosion than crystalline Fe-Cr alloys for chromium levels in the amorphous samples of greater than 2% [2,3,4]. This is attributed to the amorphous nature of the material. Amorphous material have no grain structure, and hence no grain boundaries to be attacked by corrosion. The distribution of constituent elements in the alloy is also more uniform in amorphous materials, so there are no areas which are chemically more prone to corrosive attack.

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Tsuru and Latanision [5,6] produced microcrystalline samples of a number of stainless steel alloys using the two-roller quench technique. These samples were compared to wrought samples of the same alloys. The RS samples had grain sizes of 2-5 micrometers and the wrought samples had grain sizes of 70-100 micrometers. It was found that in a chloride-free solution the general corrosion resistance of RS and wrought samples was the same, but the RS samples were more resistant to pitting corrosion. It was also noted that the RS samples may be more resistant to stress corrosion cracking than wrought samples.

Yurek et al [7] produced an RS 303 stainless steel powder by atomization in helium. The powder particles were in the range of 50-200 micrometers and were sieved to -140 mesh (approximately 100 micrometers). The grain size in this fraction was 5-10 micrometers. It was found that RS 303 was more resistant to oxidation than wrought 303, and was comparable to wrought 310, even though it has a lower chromium and nickel content than does 310.

The study described in this report was undertaken to assess the improvement in corrosion resistance of a number of RS stainless steel alloys. The alloys investigated included 304, 316 and 410 stainless steels, as well as a 304 type alloy with a reduced chromium content. This last alloy was included to assess the effect that reducing the chromium content has on the corrosion resistance.

The method used to produce the RS alloys was melt spinning. Although probably not the method to be used in pilot production or in actual production, this is a relatively simple method of producing an RS material. Pilot production would likely use an atomization procedure.

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This final report describes the details of the experimental procedures, and the analysis of the properties of the RS products. It also reviews some of the properties of commercially available, water atomized, 316 stainless steel powders. The discussion section considers a number of topics, including the importance of the chill wheel material, the corrosion resistance of RS alloys relative to wrought alloys, and the effects of reduced chromium content on the corrosion resistance of RS alloys. Finally, recommendations are made concerning the direction which further investigations could follow in development of RS stainless steel alloys.

2. MELT SPINNING

2.1 Samples

The alloys chosen for melt spinning trials were 304, 316 and 410 stainless steel alloys. Rods of each were purchased through commercial outlets. In addition, a portion of the 304 bar stock was combined with iron and nickel to produce an alloy with approximately 14% chromium content, rather than the 18% found in 304. This new alloy was melted, and ingots were made.

Sample ingots were machined from each of the above bars and ingots. These were in the form of thick walled tubes 8 mm (2 inches) long (Ref. Figs. 1 and 2). One end of each of the ingots was rounded to better fit into the round bottom crucible in the melt nozzle. The mass of a sample ingot was approximately 70 grams.

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The hole in the centre of the sample ingot allowed for the use of a stopper rod to plug the hole in the bottom of the crucible. Thus, the melt could be contained until the desired temperature and soak time were achieved. For many of the runs, the stopper rod was a hollow tube, and a thermocouple was inserted into the tube to measure the temperature of the melt.

2.2 Melt Nozzle

The melt spinning of stainless steel presents a number of technical problems. For low temperature, non-ferrous alloys, the sample can be contained in a graphite crucible. Graphite has the advantage of being easily machined, inexpensive, and readily heated by an induction heater. Unfortunately graphite is very rapidly attacked by a ferrous melt, and carbon is a serious contaminant in stainless steel. For this reason it was decided that a ceramic crucible was required. Candidate materials considered, were mullite, zirconia and alumina.

Crucibles of MV-30 mullite, in the form of tubes 3/4 inch I.D., 1 inch O.D. and 6 inches long, closed one end, were obtained from McDanel Refractories, Beaver Falls, PA. A hole approximately 3 mm in diameter was drilled in the bottom of each crucible. It was found that, after a melt spinning run, the inside of the crucible was coated in a black, glassy material, presumed to be a silica phase. The mullite crucibles broke during each run.

Enquiries were made with McDanel Refractories concerning the suitability of zirconia for the melt crucible. Zirconia has the advantage of being susceptible to induction heating, at high temperatures. Although McDanel could apply zirconia crucibles, their advice was not to use zirconia because it would be expensive (at least three times the cost of mullite or alumina) and would probably crack as readily as either mullite or alumina.

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The final choice was alumina. 998 Alumina tubes of the same dimensions as the mullite tubes were obtained from McDanel Refractories. The cost of these tubes was comparable to (and slightly less than) the cost of mullite. A hole was drilled in the bottom of each of these crucibles as well. It was found that there was no residue on the inside of the alumina tubes after a run. The alumina tubes also broke during each run, but the fractures were quite clean, and the pieces could be reassembled and reused for several runs without difficulty. The melt crucible is shown in Figure 1.

The melt crucible was contained in a quartz nozzle, also shown in Figure 1. Quartz can not be used for any extended period of time at temperatures above approximately 1200°C without the onset of recrystallization. It was found that, although the melt was usually at approximately 1570°C, the outside of the melt crucible was seldom above 1400°C, and the quartz nozzles were able to survive for many runs. Usually, they broke from repeated handling long before any noticeable recrystallization set in.

The dimension of the quartz nozzles were 28 mm I.D., 32 mm O.D. and 400 mm long. The bottom of the nozzle was rounded and closed, except for a 9.5 mm diameter hole, to allow the melt to be directed from the crucible to the wheel (ref. Fig. 2).

The molten sample was contained in the melt crucible by a stopper rod which passed through the melt to plug the outlet hole in the bottom of the crucible (Ref. Fig. 2). This rod extended up through the top of the quartz crucible. When the time came for the melt to be released onto the chill wheel, the stopper rod was raised, opening the hole at the bottom of the crucible.

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Initial trials used a quartz tube for this stopper rod, but it was found that the rod was attacked by the melt. The next material tried was alumina. 998 Alumina thermocouple tubes, 5 mm I.D., 7 mm O.D. and 600 mm long were obtained from McDanel Refractories. Although more expensive than the quartz tubes, they were found to be unaffected by the melt. They were, however, subject to cracking and often lasted for one run only. When these tubes were used, a tungsten-rhenium thermocouple was inserted into the tube to measure the temperature of the melt.

To cut down on the cost of materials, a second type of stopper rod was used also. These were rods of molybdenum. Although molybdenum has a high melting temperature (2620°C), it is attacked readily by the melt. To preserve the rods and protect the integrity of the melt, the rods were plasma sprayed with alumina. This coating was found to protect the rods for many runs. Since the rods were solid, it was not possible to use a thermocouple to measure the melt temperature. For these runs, the temperature was measured optically.

Stainless steel couples weakly to the R.F. field of the induction heater. It is thus heated by the field, but inefficiently. It was found that the full power (10 kW) of the induction heater was required to melt the sample. To make the system more efficient, a graphite insert, seen in Fig. 1., was put into the crucible, as shown in Fig. 2. This insert extended down to approximately 6 mm above the sample ingot. Since graphite couples strongly with the R.F. field, the insert became quite hot and the sample was found to melt readily at much lower power levels. It was also found that, with the graphite insert, the alumina stopper rod had less of a tendency to break on heating.

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A schematic diagram of the assembled nozzle is shown in Figure 2. The nozzle is shown in place on the melt spinning apparatus in Figure 3.

Since molten stainless steel is susceptible to attack by oxygen, the nozzle was flooded with argon gas. The quartz tube was sealed with an end cap assembly which allowed for a gas inlet, and sealed around the stopper rod. The end cap is also shown in Figure 1.

2.3 Melt Spinning

The process of melt spinning involves directing a jet of molten metal onto a spinning metal wheel. The process is very dynamic, and must be carefully controlled. One of the most important aspects is to maintain a stable molten puddle on the wheel. This puddle is the source of the melt spun strip. To maintain this stability, the wheel must be running smoothly, and have a smooth finish.

Another important aspect is that the wheel and puddle should be chemically compatible. They should not attack one another chemically, but the puddle should have some affinity for the wheel. The contact angle between the puddle and the wheel should be small, resulting in a more intimate contact between the melt and the wheel. This has two desirable effects: better thermal contact, to increase the coefficient of heat transfer; and the tendency to form thinner strips, thereby increasing the cooling rate. It is interesting to note that the thermal conductivity of the wheel, in these short runs, was found to be not of great importance.

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To better assess the importance of wheel material, a number of wheels were mounted on the melt spinning apparatus. The materials used for the wheels were mild steel (1020), stainless steel (304) and copper.

The wheel diameters were 152 mm (6 inches). The surface speed of the wheel could be adjusted up to approximately 35 m/sec.

Although kept clean, the wheel was not intentionally polished before each run, because the wheel surface was degraded from a fine polished state very early in the run, making polishing of little importance. A brass brush was pushed against the wheel during a run to help keep the wheel clean. This also helped to remove chunks of strip which may stick to the wheel. If not removed, these chunks can build up on the wheel and break the nozzle.

2.4 Melt Temperature

The melt temperature was normally brought to 1570°C. Tungsten-rhenium thermocouples placed at the bottom of the stopper tube were used to measure the sample temperature on many of the runs. The thermocouple voltage was read on a potentiometer and the temperature was determined from calibration tables supplied by the thermocouple manufacturer. In runs which used a solid stopper rod, the temperature was measured by means of an optical pyrometer.

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Since alumina is not heated by induction, all heat was generated from inside the melt crucible, with the result that a temperature gradient developed between the inside and the outside of the crucible. On temperature calibration runs, it was found that when the melt temperature was 1570°C (as measured by thermocouple), the temperature on the outside of the crucible was approximately 1400°C (measured optically). The temperature on the outside of the crucible was not very sensitive to the temperature of the melt.

2.5 Procedure

The melt nozzle was assembled, as in Figure 2, and was installed on the melt spinning machine, as shown in Figure 3. This nozzle was positioned over the wheel. The variables in this positioning are: the wheel-nozzle distance, the distance from the wheel surface to the bottom of the melt crucible; the advance from perpendicular, the horizontal distance from the centre of the wheel to the hole in the melt crucible; and the angle of the inclination, the angle between the axis of symmetry of the nozzle and the vertical.

When the nozzle was positioned, the induction heater was turned on and Argon gas was flushed through the nozzle at approximately 1 psi. At this point, the wheel was stationary. To avoid heating the wheel, the nozzle was swivelled off to one side. Upon reaching the desired temperature (measured by either a thermocouple or optical pyrometer), the sample was allowed to soak for five to ten minutes to ensure a uniform temperature.

Ejection onto the wheel required a number of steps. The argon pressure was increased to 4 psi and the wheel was brought to the desired speed. The nozzle was swung back over the wheel, and the induction heater was turned off (to eliminate the sparking which often occurs between the nozzle and the wheel). The O-ring sealing the stopper rod was loosened, and the stopper rod was raised, releasing the melt onto the wheel.

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The chilled strip was thrown from the wheel by centrifugal force, and collected in a tray placed in front of the apparatus.

The experimental parameters are summarized in Table 1.

3. PROPERTIES OF STRIPS

3.1 Cleaning and Passivating

The melt spun strips were found to be coated with what appeared to be an oxide layer. Some were cast under a flowing argon cover, but this did not affect the degree of oxidation. As a result, it was decided to clean the strips chemically.

The method used to clean and passivate the strips was that recommended in the ASM Metals Handbook, 9th ed., Volume 5. They were descaled by immersion in a solution of 20 volume % nitric acid, and 2 volume % hydrofluoric acid for approximately 15 minutes.

Following cleaning, the strips were passivated. Stainless steel derives its corrosion resistance from a surface layer of protective oxides. Although these oxides will form naturally, a more uniform coating can be formed by oxidizing the surface chemically. This process is called passivating. The strips were passivated by immersion in a solution of 70 volume % nitric acid for 30 minutes.

3.2 Physical Properties

The properties measured included strip width and thickness (before and after cleaning and passivating), chemical composition, grain size distribution and microhardness. Strip width was not affected by cleaning, but the thickness was

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changed on many of the samples. Strip dimensions are given in Table II. Strip widths are typically between 4 and 9 mm, and strip thicknesses are approximately 0.1 mm.

All strips were analysed for chemical composition. The results of these analyses are shown in Table III. The composition of the low chromium sample was verified to be 14%. None of the samples were contaminated with carbon by the presence of the graphite inserts used to aid in heating.

Portions of each strip were mounted for metallographic examination. Photographs of all strips are shown in Figures 4 through 7. Samples of wrought materials were mounted also. Microstructures of these samples are shown in Figures 8 through 11.

The grain size distribution was measured for each sample, both wrought and melt spun. The results of these measurements are shown in Table IV. The standard deviations of the grain size distributions are also given in Table IV.

In the 304 and 316 samples, the RS structure was approximately 2.5 times smaller than that of the corresponding wrought material. The effect on the 410 was not obvious, since the wrought 410 had a very fine microstructure already. The low chromium sample has a poorly defined microstructure in the cast form, but the grain size in the RS form was comparable to that of the RS 304 and 316.

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Hardness measurements were made on all samples, and these results are also shown in Table IV. Hardnesses were measured on cross sections of the ribbons. There was no significant increase in the hardness of rapidly solidified 304 or 316 over that of wrought samples. This is consistent with the observation that these alloys are not quench hardenable. However, there was a noticeable increase in the hardness of the 410 and the low chromium rapidly solidified samples over that of the wrought samples.

3.3 Corrosion Resistance

All melt spun products were subjected to corrosion resistance testing. The tests chosen were i) ASTM G61-78, the test for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion, and ii) ASTM A262-84 Practice B, a test for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels using boiling ferric sulphate-sulphuric acid. ASTM G61 is sensitive to pitting or crevice corrosion, and ASTM A262 determines sensitivity to intergranular corrosion.

Samples were prepared for ASTM G61 (Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion). Pieces of ribbon 18 cm long were cut from each sample. A 2.5 cm long section at one end was polished on both sides to 600 grit using a hand-held electric polishing tool. The samples were each placed in a piece of glass tubing (8 mm ID), 13 cm long and sealed in place using an electrochemically inert resin so that the polished portion of the sample protruded from one end and an unpolished portion of the sample protruded from the other end of the tubing.

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The technique consists of producing a part of a potentiodynamic anodic polarization curve in 3.56 weight percent NaCl at $25 \pm 1^{\circ}\text{C}$. Instead of completing the curve however, the scan direction is reversed when the current density reaches 5.0 mA/cm^2 . The scan rate is 0.6 volts per hour throughout the experiment.

Figure 12 shows two representative scans from the ASTM G61 specifications. One scan is for 304 SS which shows susceptibility to localized (pitting or crevice) corrosion and the other scan is for Hastelloy C276 which shows resistance to localized corrosion. The important feature of these curves is the potential (known as the protection potential) at which the reverse scan crosses the forward scan relative to the corrosion potential. In the standard, the reverse scan for 304 SS crosses the forward scan at the corrosion potential. Therefore, the material would be susceptible to pitting corrosion at any potential above the corrosion potential. The cross-over for Hastelloy C276, on the other hand, is well above the corrosion potential indicating that C276 could be polarized some 800 mv positive to the corrosion potential without localized corrosion occurring.

The results from this test on the RS samples, and on a sample of wrought 304 stainless steel are shown in Table V. The chemical composition of the wrought 304 sample is given in Table III.

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Potential vs current curves for all samples, as measured by the method of ASTM E51, are shown in Figures 13 through 17. The critical pitting potential, E_c , and the protection potential, E_p , are shown in each plot. Pits initiate at potentials positive to E_c and propagate at potentials between E_c and E_p . At potentials negative to E_p pits repassivate and cease to grow.

The ribbons were very thin which made polishing and handling difficult. The ease with which the samples for the electrochemical test could be bent may affect the results by making local areas more susceptible to corrosion because of cold work. Polishing the samples (to remove depressions in the surface which could act as pit initiation sites) was difficult without perforating the samples. The samples were free from perforations before being tested in the electrochemical tests.

In addition to the electrochemical test, all samples except the 304 sample melt-spun on the steel wheel were subjected to the ASTM A262 Practice B test (boiling ferric sulphate-sulphuric acid test). The corrosion rates and comments on the performance of these materials are shown in Table VI.

It was difficult to find a large enough sample free from perforations to perform the chemical test. Because of the perforations and the difficulty in polishing, the samples for the chemical test were not polished.

The 304 SS appears to have some resistance to localized attack. The 316 SS may have perforated due to mechanical action at thin areas rather than as a result of localized

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attack. This would be consistent with the electrochemical test which showed a relatively high general corrosion rate and good resistance to localized attack. The 410 SS corroded very severely especially at the dark centre region. The low Cr sample was also perforated at thin areas due to mechanical action rather than as a result of localized attack.

3.4 Properties After Heat Treating

It is expected that the consolidation of RS materials may involve some type of a sintering process to produce a final component. To aid in determining the conditions for this sintering process, the thermal stability of the material should be known.

Portions of all RS strips were subjected to annealing for one hour in a hydrogen atmosphere at temperatures of 600, 800, 1000 or 1200°C. Heat treated samples were mounted for metallographic examination. The microstructures of these samples are shown in Figures 18 through 22.

The microhardness of all samples was measured. The results are listed in Table VII and shown in Figure 24. The 300 series alloys were not significantly affected by the heat tests. The low chromium alloy showed a general trend of decreasing hardness across the temperature range investigated. The 410 samples showed a marked variation with temperature, in the form of a significant decrease in hardness at higher temperatures.

The grain size distribution for all samples was measured also. Results are listed in Table VIII and shown in Figure 25.

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Again, the 300 series alloys are not significantly altered by the heat treatment, although there is a definite trend to larger grain sizes at higher temperatures. The 410 samples showed a higher degree of grain growth, especially for temperatures above 1000°C.

It should be noted that some of the as melt-spun samples displayed grain sizes larger than samples after heat treatment. This phenomenon is the result of variable grain size within the melt spun strip.

The above observations indicate that these RS materials are able to withstand a reasonable amount of annealing (up to approximately 1000°C) without significant alteration to their physical properties.

4. PROPERTIES OF COMMERCIAL ATOMIZED POWDERS

There are a number of commercially available, water atomized stainless steel powders. The water atomization process produces fine powders which may have cooling rates high enough to produce RS materials. The powders were obtained from Metallurgical Industries Inc., of Tinton Falls, New Jersey, from Pfizer Minerals, Pigment and Metals Division, Pennsylvania and from Hoeganaes Corporation, New Jersey.

The powder obtained from Metallurgical Industries was a 316L Injection Moulding Powder, of 10 micron size, with a Fisher Sub Sieve Size of 11.2 microns. The sample received was from Lot AB46118. The chemical composition of the sample is given in Table IX.

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The powder obtained from Pfizer was P316L Super-Fine Injection Moulding Minus 30 Micron size powder. The sample received was from Lot 039285. The chemical composition of this sample is also given in Table IX.

The powder obtained from Hoeganaes was Ancor 316L. The sample received was from Lot 12-46426. The mesh size distribution of this powder is: -80/+100 - 2%, -100/+325 - 56%, -325 - 42%. The chemical composition is given in Table IX.

Samples of all three powders were mounted for metallographic examination. The microstructure of the Metallurgical Industries powder is shown in Figure 26, that of the Pfizer powder is shown in Figure 27, and that of the Hoeganaes powder is shown in Figure 28.

Grain size distribution measurements were carried out on all powders. Forty grain size measurements were made for each powder, and the mean and standard deviation of the grain size was determined. The powder from Metallurgical Industries had a mean grain size of 2.04 micrometers with a standard deviation of 0.92 micrometers. The powder from Pfizer had a mean grain size of 2.01 micrometers with a standard deviation of 0.91 micrometers. The powder from Hoeganaes had a mean grain size of 4.13 micrometers with a standard deviation of 1.1 micrometers. These are very fine grain size distributions for 316 samples (compared to the grain size of 14 micrometers in the melt spun sample listed in Table IV).

Based on grain size measurements alone, these commercially available water atomized stainless steel powders appear to be Rapidly Solidified. However, the inherent high surface oxygen content of water atomized powders may affect corrosion properties.

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5. DISCUSSION

In this discussion, three aspects of RS stainless steel are considered. These aspects are wheel composition, corrosion resistance and chromium content.

5.1 Wheel Composition

The process of melt spinning is not well understood. The mechanism of heat extraction from the melt is a point of debate. There are two limiting regimes which theories are able to describe. These regimes are described as Newtonian and Ideal [8,9]. In Newtonian cooling, temperature gradients in the melt and the wheel (substrate) are negligible. There is, however, a large discontinuity in temperature at the interface between the melt and the substrate. The cooling rate is controlled by the coefficient of heat transfer and is not dependent on the thermal conductivity of the melt or substrate. In Ideal cooling, temperature gradients exist in both the melt and the substrate, with no temperature discontinuity across the interface between the melt and the substrate.

There has been some evidence put forth to support the claim that the cooling in melt spinning takes place in the Ideal cooling regime [10]. In the melt spinning experiments described in this report, the 304 and 410 alloys were cooled on a steel and a stainless steel wheel, (The thermal conductivity of mild steel is $51.9 \text{ W/m}^{\circ}\text{C}$ and the thermal conductivity of stainless steel is $17.3 \text{ W/m}^{\circ}\text{C}$). In both alloys, the grain size was slightly smaller and the hardness slightly higher in the samples chilled on the steel wheel. This indicates that the thermal conductivity of the substrate is of some importance in the cooling process, implying that the process tends to be in the Ideal cooling regime. The effect is not pronounced.

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Even though the samples produced on a stainless steel wheel had a somewhat lower cooling rate, they were clearly Rapidly Solidified, and the choice of a stainless steel wheel may be advantageous, because of the corrosion resistance of the wheel, for large scale production of RS strip.

Although cooling rates were not measured in this project, estimates can be made of the cooling rate. An examination of Table I shows that a typical melt spinning wheel speed is between 10 and 30 m/sec. The strip is observed to be in contact with the wheel over a distance of approximately 5 cm. Thus the amount of time spent on the wheel is between 1 and 5 milliseconds. Table I indicates also that the sample temperature is between 50 and 100°C above the melt temperature. If the temperature of the melt is to drop below the melting temperature during the time that the strip is in contact with the wheel, the cooling rate must be between 10,000 and 100,000 K/sec. These cooling rates are regarded traditionally as RS.

5.2 Corrosion Resistance

The resistance to localized corrosion in Rapidly Solidified stainless steel strips appears to be improved over that in wrought samples. This indicates that some aspects of corrosion resistance are improved by Rapid Solidification.

Furthermore, this investigation shows that commercial water atomized stainless steel powder have been Rapidly Solidified. This represents a source of significant quantities of Rapidly Solidified stainless steel. However, the oxygen content of these powders might be detrimental to the corrosion resistance of products made from them.

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One outcome of this investigation is that the RS properties of stainless steels are able to survive some level of heat treatment. This is an indication that one method of improving the corrosion resistance properties of stainless steel P/M components may be to start with a fine, water atomized powder. This powder would be compacted and then sintered using a process designed to bond the powder particles, but not significantly degrade the RS properties.

5.3 Chromium Content

One aspect investigated in this study was the effect of reducing the chromium content in the alloy. A reduced chromium alloy was made by adding iron and nickel to a commercial 304 alloy. This had the effect of reducing the chromium level from 18% to 14%. (See Table III for the actual chemical compositions). It was found that the susceptibility to localized corrosion in the reduced chrome alloy was increased over that of the RS 304 alloy. However, the RS reduced chrome sample was found to be comparable to wrought 304. Thus Rapid Solidification may be economically feasible in cases in which reducing chromium levels (without loss in corrosion resistance) would represent a considerable cost saving in raw materials.

6. CONCLUSIONS

This investigation has shown that RS stainless steel is more resistant to localized corrosion than is wrought stainless steel of the same composition.

The reduced chromium RS stainless strip at least as corrosion resistant as a wrought sample of the conventional alloy. This raises the possibility of using reduced chromium content alloys without the loss of corrosion resistance.

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The microstructures of commercially available water atomized stainless steel powders are comparable to those of RS materials, (and were, in fact, finer than those of the melt spun strips produced in this project).

RS stainless steel materials can withstand at least some level of heat treatment without significant loss of RS properties. The temperatures of this heat treatment can be as high as 1000°C and could last an hour or more.

The commercialization of RS stainless steel requires that there be a source of RS materials, and a method of producing a bulk component from these materials. Since at least one dimension of an RS material (produced by rapid chilling) must be small, the production of a bulk component will require some form of consolidation. This may involve a carefully controlled sintering program, possibly coupled with some unique method of powder compaction. P/M components are susceptible to corrosive attack in the regions between the original powder particles. This inherent weakness must be considered in the assessment of the commercialization of RS materials.

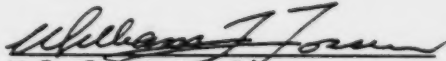
7. RECOMMENDATIONS

1. A more detailed assessment of the effects of reducing the chromium levels of stainless steels on their corrosion resistance should be carried out.
2. Alternate alloys containing, for example, aluminum, silicon or manganese should be investigated using RS techniques.

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3. Methods should be investigated for the consolidation of RS powders which do not degrade the RS properties of the powders. Such methods may include hot extrusion, cold compaction and sintering, or more exotic compaction processes.
4. Since water atomized powders tend to have high surface oxygen levels, alternate fine powder manufacturing processes should be considered.



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:lu
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TABLE I

EXPERIMENTAL PARAMETERS

Alloy	Wheel Composition	Wheel Speed (m/sec.)	Nozzle Diameter (mm)	Wheel-Nozzle Distance (mm)	Advance from Perpendicular (mm)	Angle of Inclination (degrees)	Ejection Temperature ($^{\circ}\text{C}$)
304	Steel	17	2.5	2	15	5	1490 (Optical 1320)
304	Stainless Steel	12	3.7	2	3	5	1570
316	Steel	22	3.7	2	3	5	(Optical 1400*)
410	Steel	31	3.7	6	4	7	(Optical 1400)
410	Stainless Steel	17	3.7	2	3	7	(Optical 1400)
"Low Chrome"	Steel	31	3.7	6	4	7	1540

* An optical reading of 1400°C on the outside of the alumina crucible corresponds to a thermocouple reading of 1570°C in the melt.

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TABLE II.

Machine parameters and strip dimensions
for melt spun stainless steel alloys

<u>Alloy</u>	<u>Wheel Type</u>	<u>Wheel Speed (m/sec.)</u>	<u>Strip Width (mm)</u>	<u>Strip Thickness (mm)</u>	
				<u>Before Descaling</u>	<u>After Descaling</u>
304	Steel	17	5	0.14	0.09
		12	8	0.10	0.095
	Stainless Steel				
316	Steel	22	8	0.09	0.09
410	Steel	31	4 to 8	0.12	0.068
		17	9	0.13	0.13
	Stainless Steel				
Low Chrome	Steel	31	5	0.088	0.088

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TABLE III.

Chemical Compositions of RS Stainless Steel Strips

<u>Alloy</u>	<u>Wheel Composition</u>	<u>Ni</u>	<u>Cr***</u>	<u>C****</u>	<u>S****</u>
304	Steel	10.1*	18.4	0.031	0.029
304	Stainless	9.7*	18.2	0.057	0.029
304W	-	9.12*****	18.15	0.042	0.003
316	Steel	10.7*	16.6	0.053	0.004
Low Chrome	Steel	9.9*	14.0	0.075	0.026
410	Steel	0.22**	12.6	0.14	0.004
410	Stainless	0.22**	12.7	0.11	0.004

* Dimethylglyoxime (Photometric) ASTM E30-77 (Reapproved 1980)

** Atomic Absorption ASTM E663-78

*** Perchloric Acid Oxidation

**** LECO Fusion Technique for Sulphur and Carbon

***** Sample used for electro-chemical test.

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TABLE IV.
Properties of Stainless Steel Samples

<u>Alloy</u>	<u>Wheel Material</u>	<u>Grain Size</u> <u>μm</u>	<u>Hardness</u> <u>(VHN-50gf)</u>
304	Steel	12 (5)*	241 (21)*
	Stainless Steel	14 (7)	184 (10)
304-Wrought		32 (12)	197 (4)
316	Steel	14 (6)	205 (16)
316-Wrought		36 (27)	233 (12)
410	Steel	7.4 (4)	535 (12)
	Stainless Steel	17 (5)	450 (47)
410-Wrought		6.6 (3)	281 (8)
Low Chrome	Steel	14 (5)	252 (53)
Low Chrome-wrought		111 defined	188 (11)

* Bracketed values give the standard deviations of the measured quantities.

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TABLE V

Cyclic Potentiodynamic Polarization
Measurements for Localized Corrosion

Material	$E_{\text{corrosion}}$	$E_{\text{protection}}$	$E_{\text{critical pitting}}$	Comments
304	-0.156	-0.132	-0.039	Shows better resistance to localized corrosion than suggested by the ASTM specification
304 SS*	-0.115	-0.015	+0.076	Shows better resistance to localized corrosion than suggested by the ASTM specification and that shown by RS 304 above.
304 W**	-0.278	-0.060	+0.130	
316	-0.296	-0.195	-0.178	Corrosion current was high for this sample indicating a high general corrosion rate. The cross-over, however, occurred very close to the highest potential reached on the forward scan, similar to the curve for C276 in the ASTM Spec. This implies a high resistance to localized corrosion.

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TABLE V continued

Cyclic Potentiodynamic Polarization
Measurements for Localized Corrosion

Material	E _{corrosion}	E _{protection}	E _{critical pitting}	Comments
410 SS*	-0.410	-0.315	-0.016	Shows resistance to localized corrosion comparable to that shown by RS 304 SS above.
Low Cr	-0.468	-0.160	-0.052	Shows a resistance to localized attack intermediate between the ASTM specifications for 304 and C276.

* Melt spun on a stainless steel wheel.

** Wrought sample.

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TABLE VI

Susceptibility to Intergranular Attack

Material	Corrosion Rate (mpy)	Comments
304 SS*	38	No apparent pits
304 W**	32	
316	51	Some longitudinal perforations.
410 SS*	2160	Centre strip corroded away; severe general attack.
Low Cr	140	Many longitudinal and transverse perforations.

* Melt spun on a stainless steel wheel.

** Wrought sample.

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TABLE VII
Microhardness of Annealed Strips (VHN)

<u>Annealing Temp (C)</u>	<u>Low Cr</u>	<u>304</u>	<u>304S*</u>	<u>316</u>	<u>410</u>	<u>410S*</u>
600	206(8)**	165(12)	172(4)	158(8)	141(10)	202(4)
800	153(8)	154(18)	146(12)	163(10)	197(9)	175(36)
1000	169(3)	151(12)	137(11)	163(17)	94(15)	108(28)
1200	122(14)	182(22)	149(19)	147(11)	130(3)	87(23)

* The suffix S denotes samples melt spun on a stainless steel wheel.
 ** Bracketed values give the standard deviations of these measurements.

TABLE VIII.
Grain Size Measurements on Annealed Strips (microns)

<u>Annealing Temp (C)</u>	<u>Low Cr</u>	<u>304</u>	<u>304S*</u>	<u>316</u>	<u>410</u>	<u>410S*</u>
600	6.90(4.8)**	6.10(3.4)	14.40(9.8)	6.90(4.4)	4.60(2.2)	4.55(1.9)
800	4.75(1.4)	6.95(2.7)	5.50(2.0)	6.35(2.8)	2.85(1.4)	5.90(3.4)
1000	7.15(2.3)	9.15(3.8)	6.40(2.3)	10.45(4.4)	7.85(4.5)	12.85(4.7)
1200	13.57(4.5)	10.30(3.4)	9.05(3.0)	11.90(6.0)	24.00(12.9)	27.80(10.0)

* The suffix S denotes samples melt spun on a stainless steel wheel.
 ** Bracketed values give the standard deviations of the measured quantities.

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TABLE IX

Chemical Compositions of Commercial Powders

<u>Powder</u>	<u>Ni</u>	<u>Cr</u>	<u>C</u>	<u>Si</u>	<u>Mo</u>	<u>Mn</u>	<u>Fe</u>
316L*	13.40	16.80	0.02	0.88	2.36	0.10	Bal.
P 316L**	12.00	17.00	0.30	0.80	2.50	--	Bal.
316L***	13.00	17.50	0.03	00.85	2.20	--	Bal.

* Supplied by Metallurgical Industries Inc., Tinton Falls, NJ.
Lot A846118.

** Supplied by Pfizer Inc., Pennsylvania. Lot 039285.

*** Supplied by Hoeganaes Corp, New Jersey. Lot 12-46426.

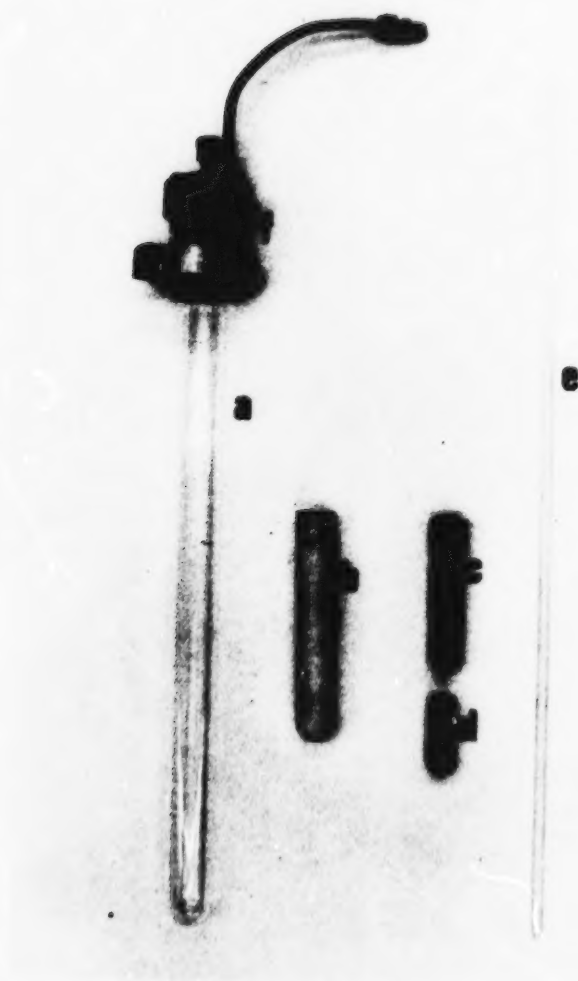


Figure 1.

Components of melt spinning nozzle, showing

- a) quartz nozzle, b) alumina crucible, c) graphite insert,
d) sample ingot, e) alumina stopper rod, & f) cap assembly.

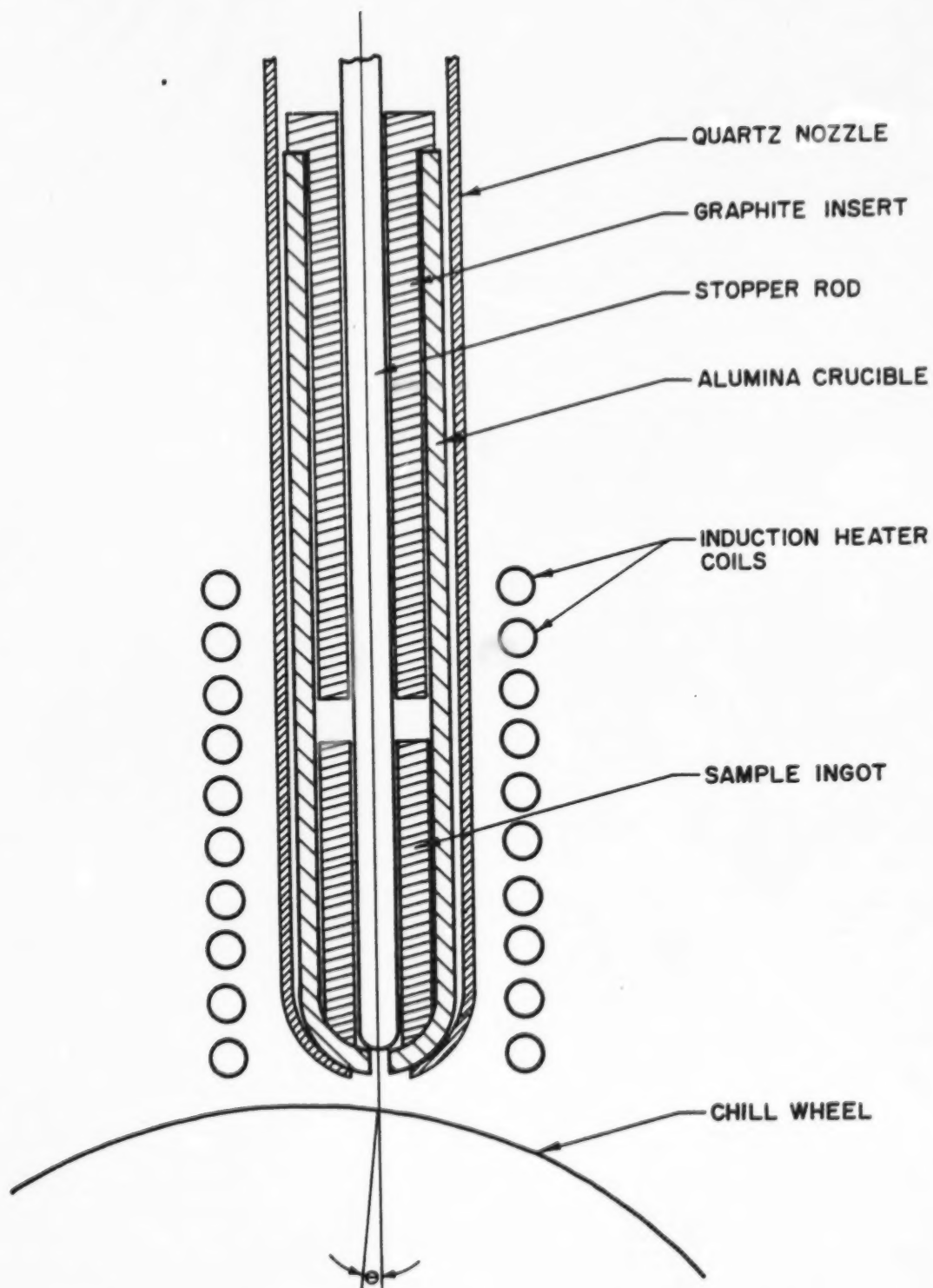


Figure 2.
Melt spinning nozzle assembly.

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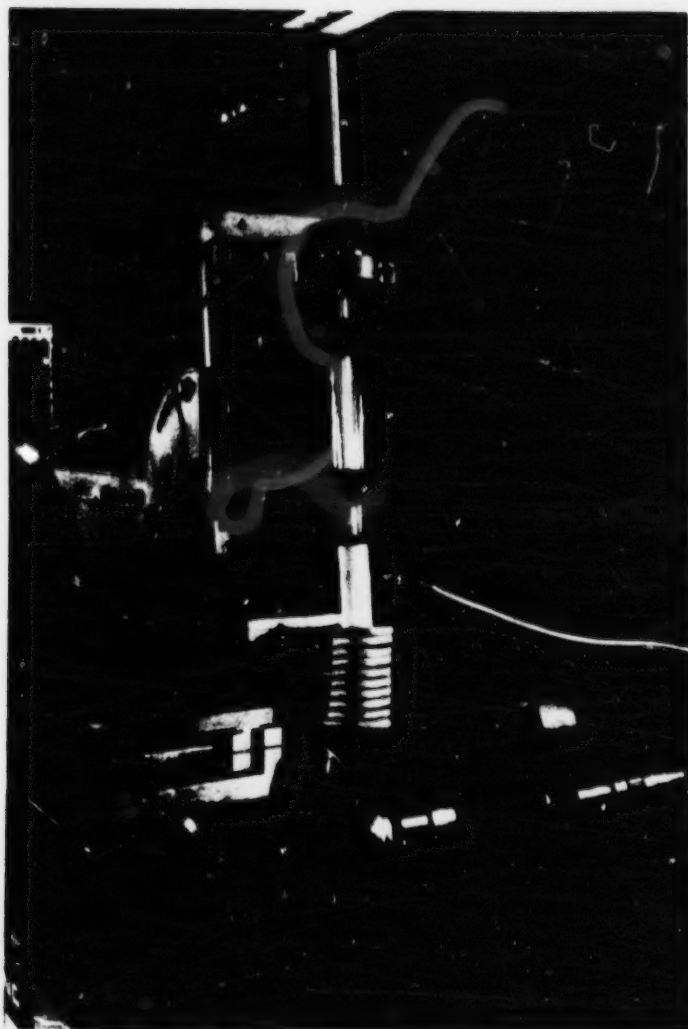
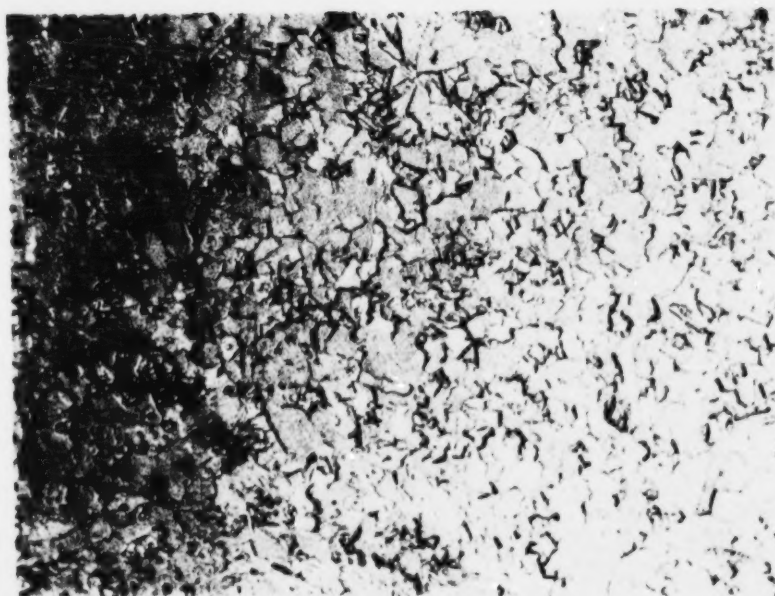
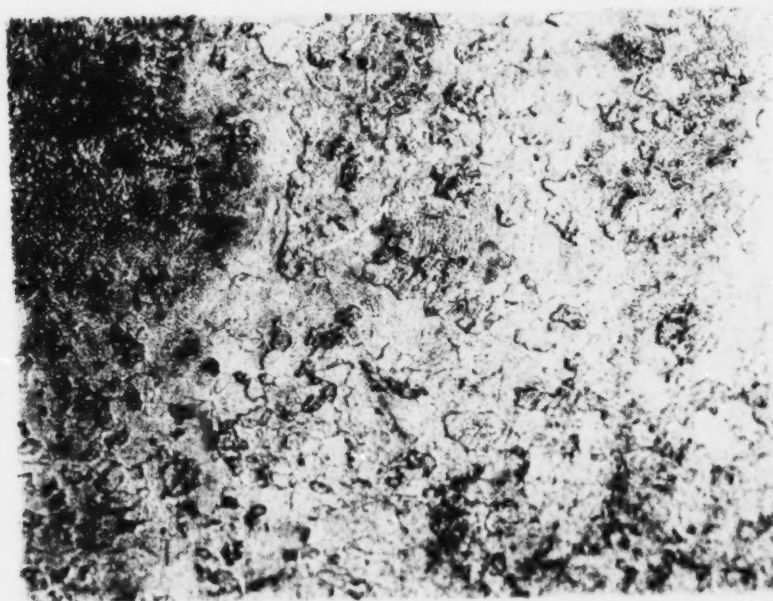


Figure 3.

Melt spinning facility with nozzle assembly in place.



(a)



(b)

Figure 4. Microstructure of melt spun 304 stainless steel (X250). Sample in (a) was chilled on a steel wheel, sample in (b) on a stainless steel wheel.

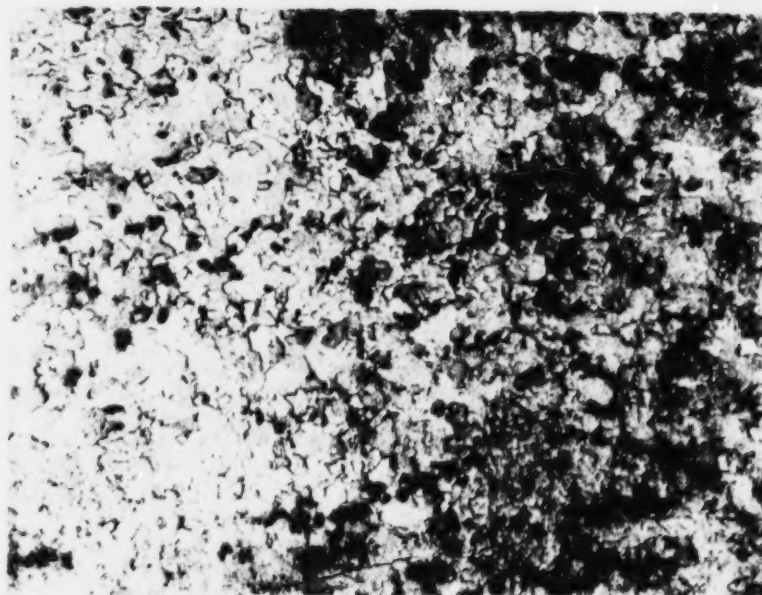


Figure 5. Microstructure of melt spun 316 stainless steel,
chilled on a steel wheel (X250).

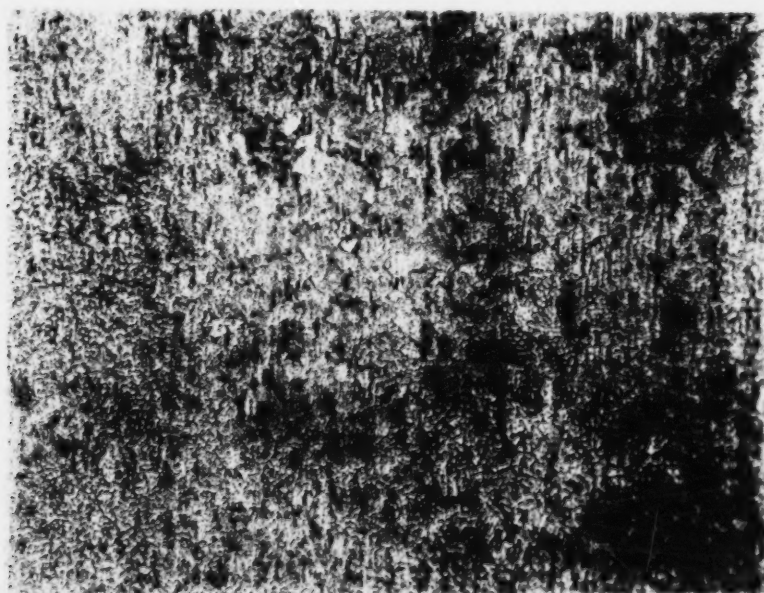
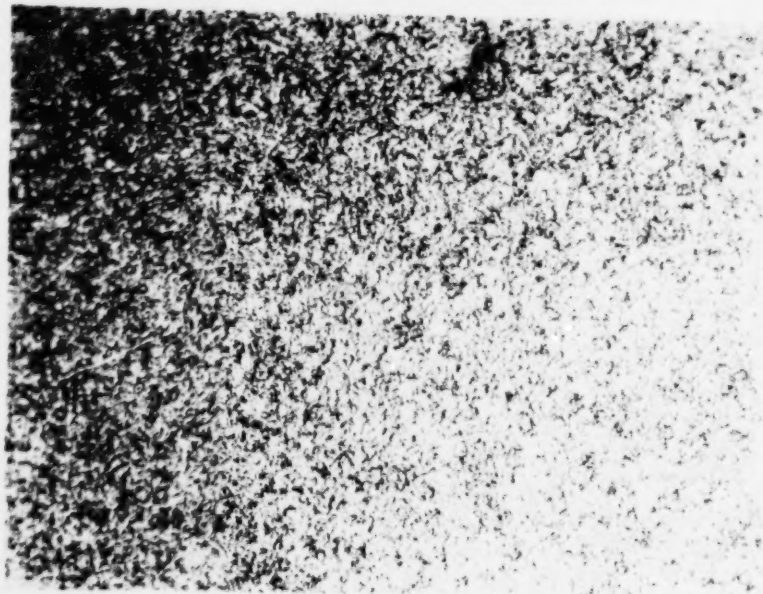
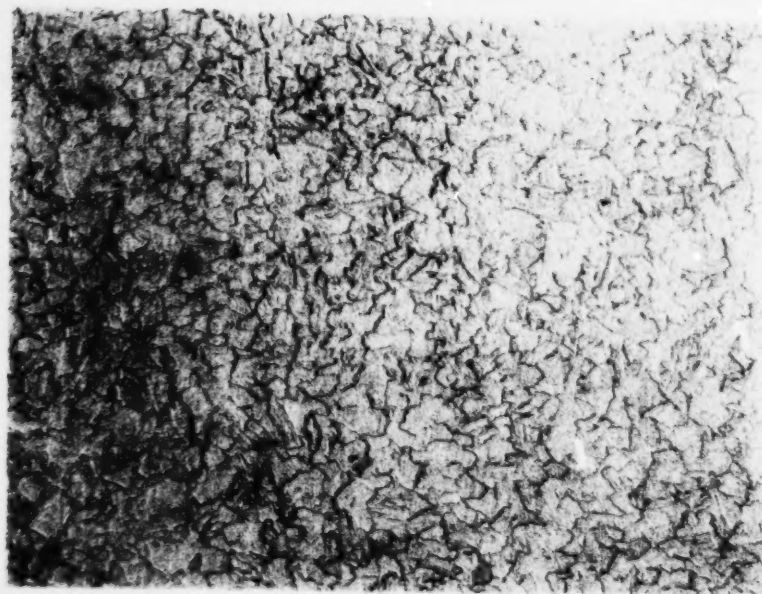


Figure 6. Microstructure of melt spun, low chromium
stainless steel, chilled on a steel wheel (X250).



(a)



(b)

Figure 7. Microstructures of melt spun 410 stainless steel (X250). Sample in (a) was chilled on a steel wheel, sample in (b) on a stainless steel wheel.

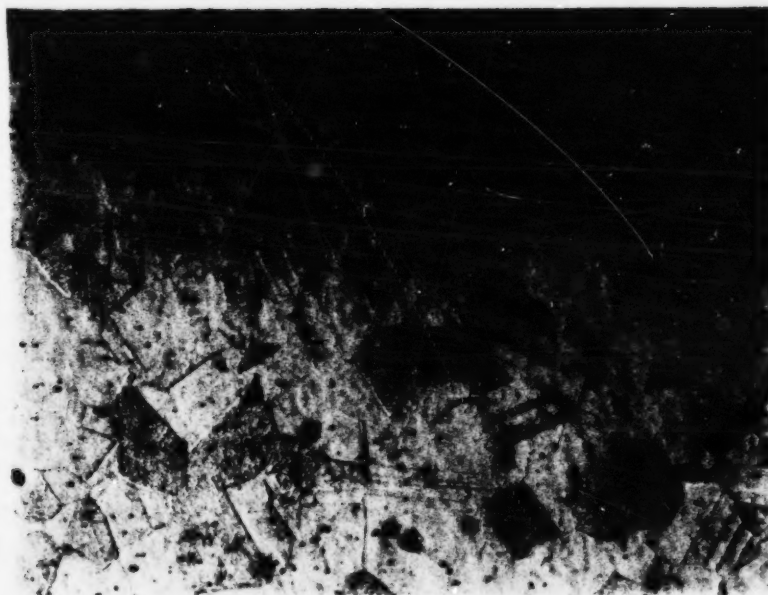


Figure 8. Microstructure of wrought 304 stainless steel
(X250).

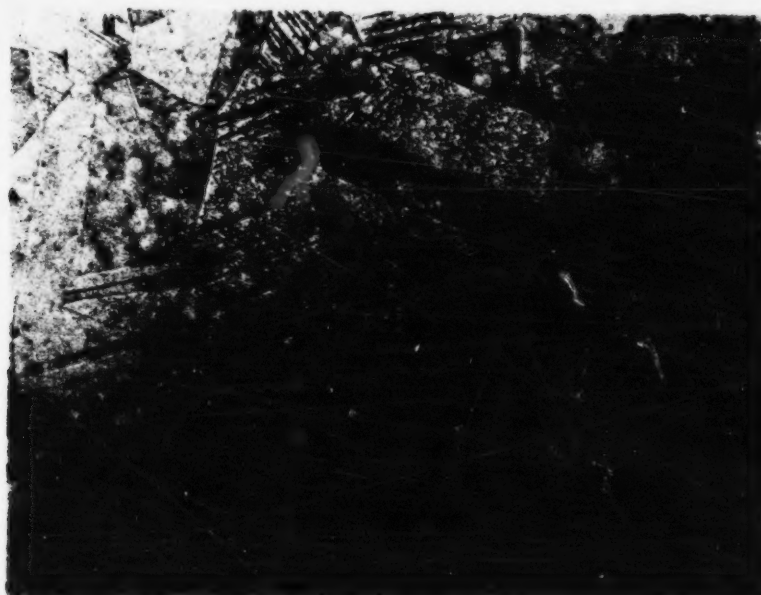


Figure 9. Microstructure of wrought 316 stainless steel
(X250).



Figure 10. Microstructure of wrought, low chromium stainless steel (X250).

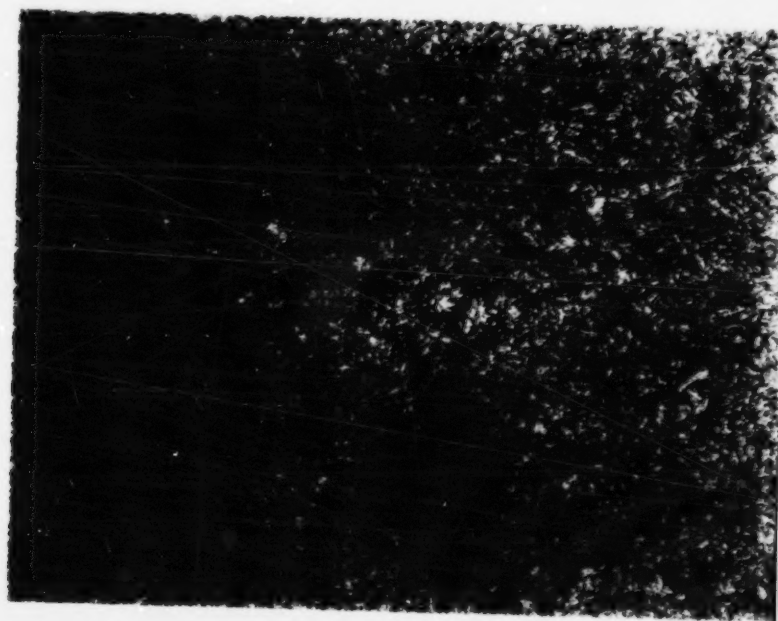


Figure 11. Microstructure of wrought 410 stainless steel (X250).

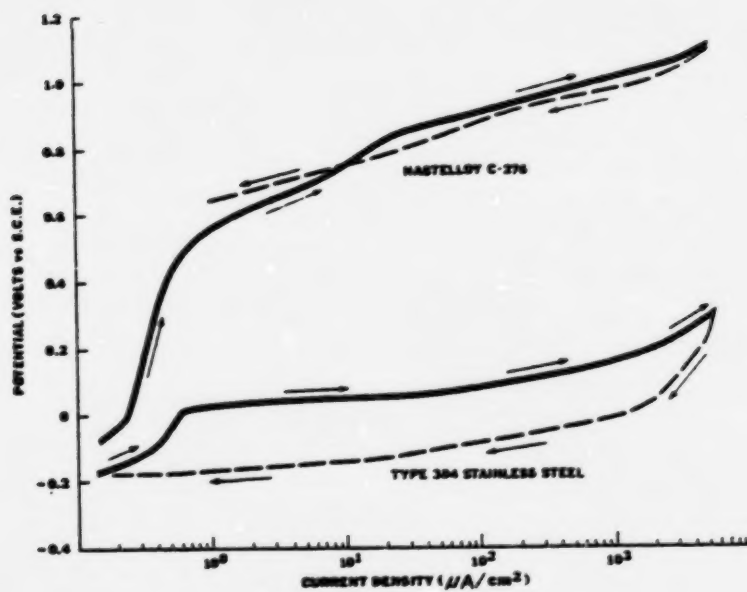


Figure 12. Representative Potentiodynamic
polarization curve from ASTM 661.

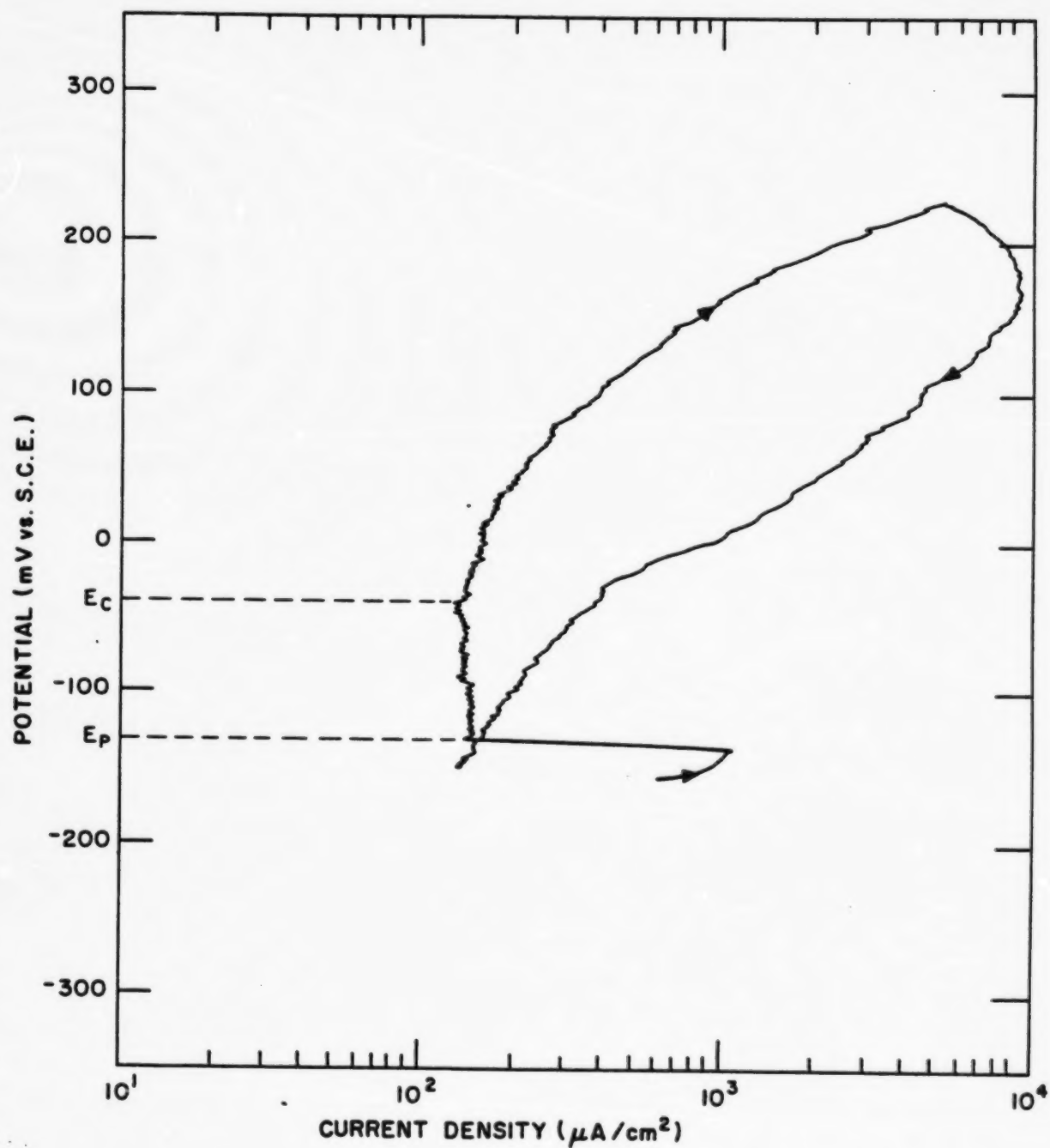


Figure 13. Cyclic Potentiodynamic Polarization curve for RS 304
Melt spun on a steel wheel.

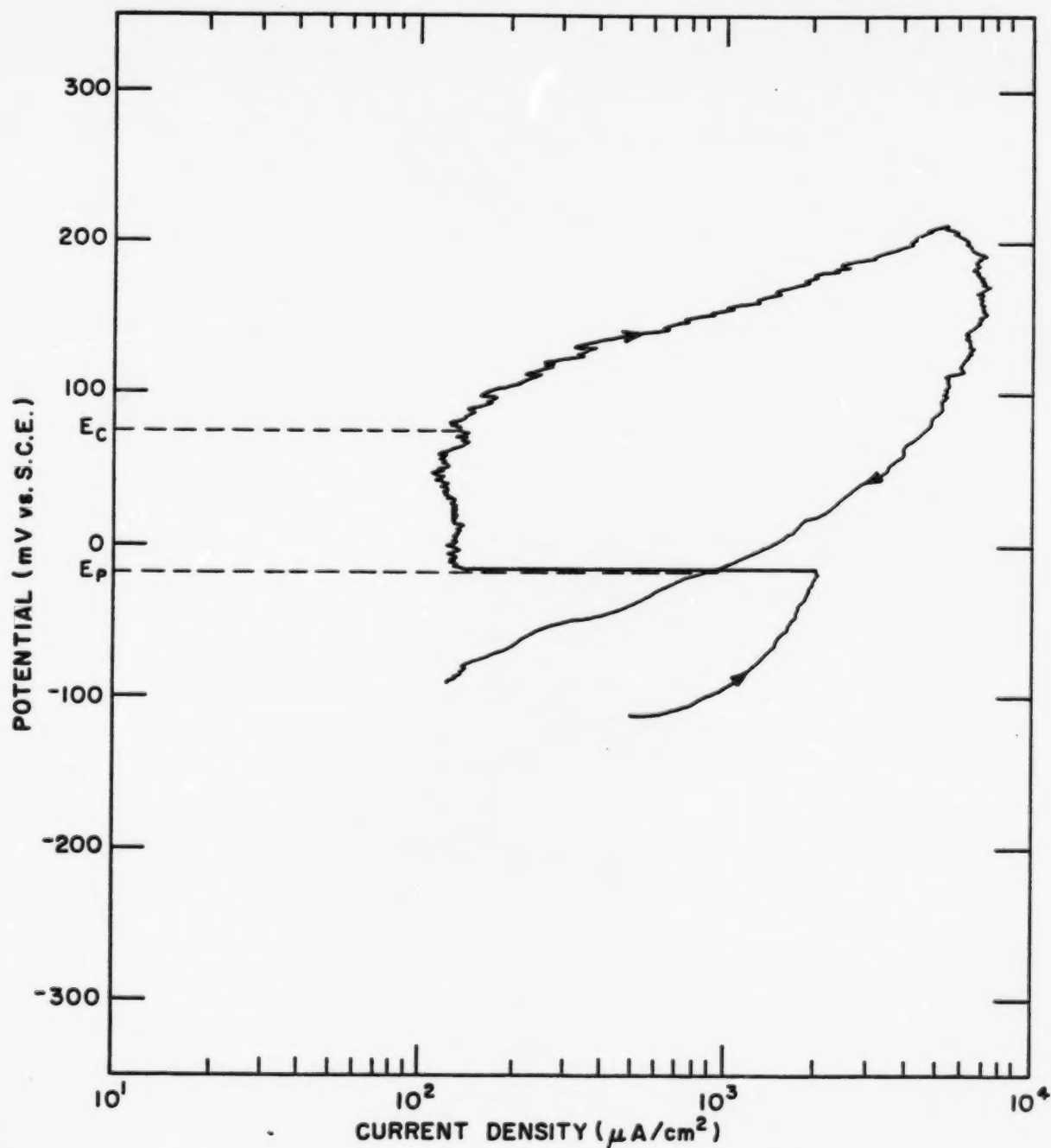


Figure 14. Cyclic Potentiodynamic Polarization curve for RS 304, melt spun on a stainless steel wheel.

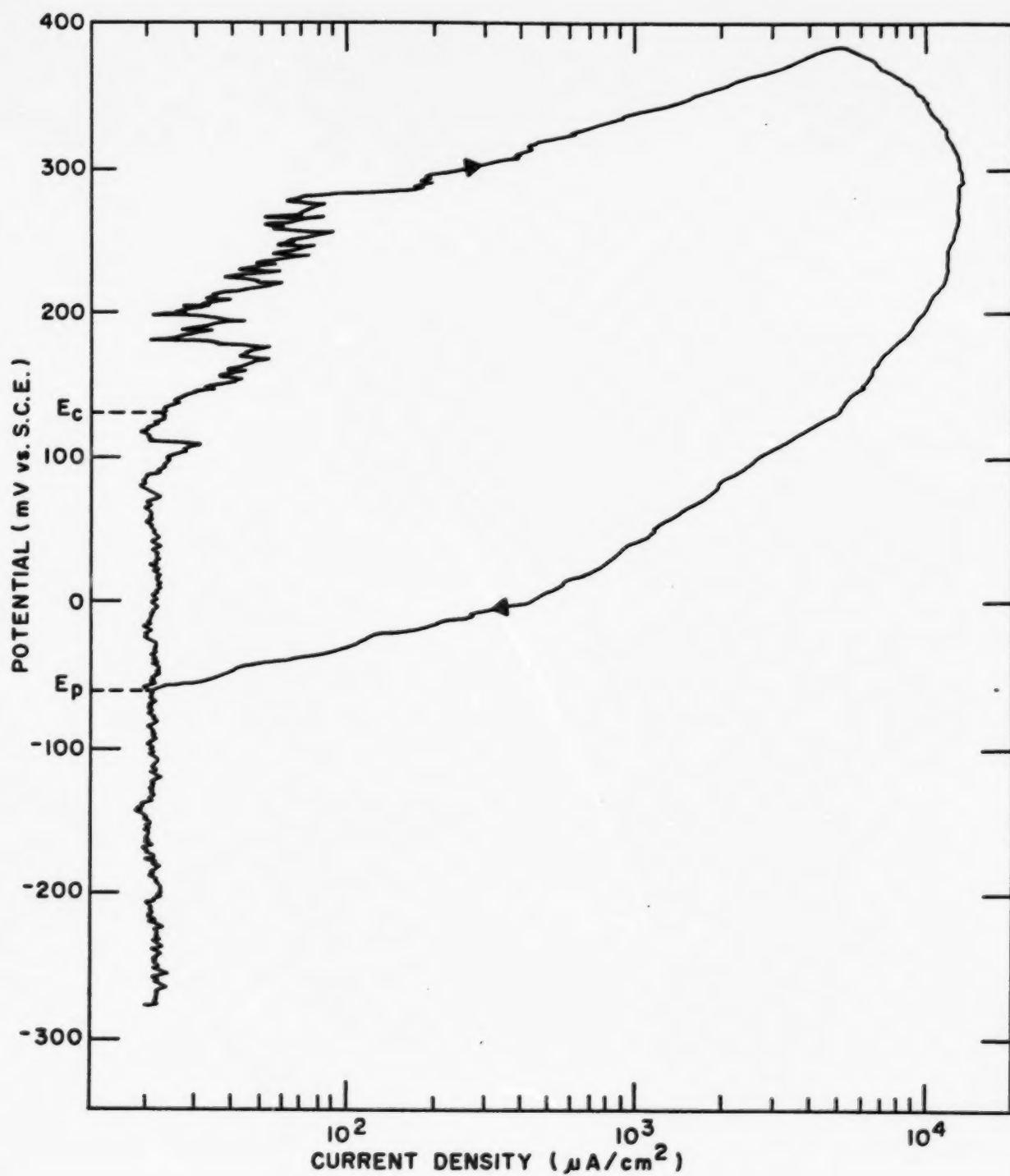


Figure 15. Cyclic Potentiodynamic Polarization curve for wrought 304.

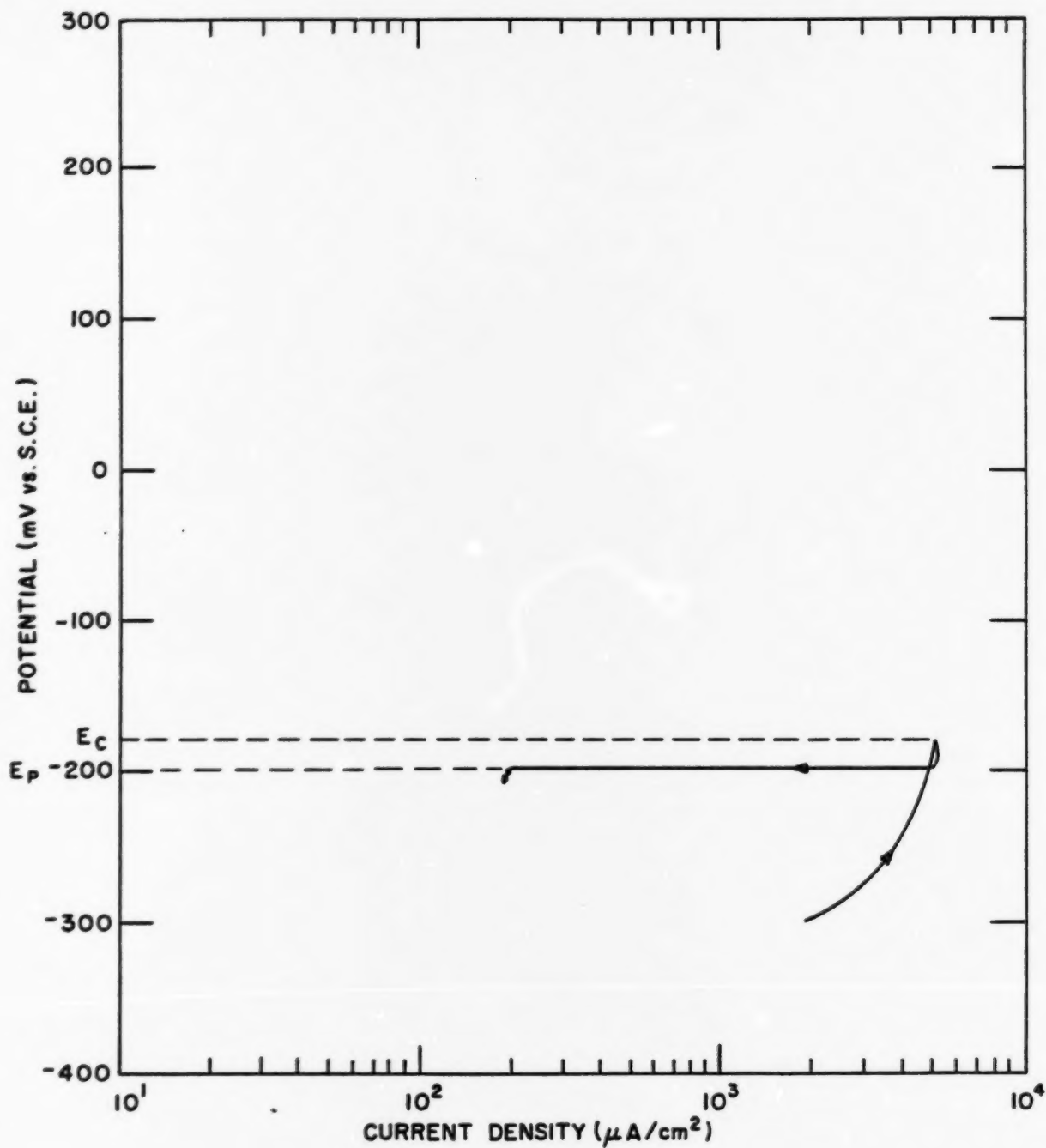


Figure 16. Cyclic Potentiodynamic Polarization curve for RS316, melt spun on a steel wheel.

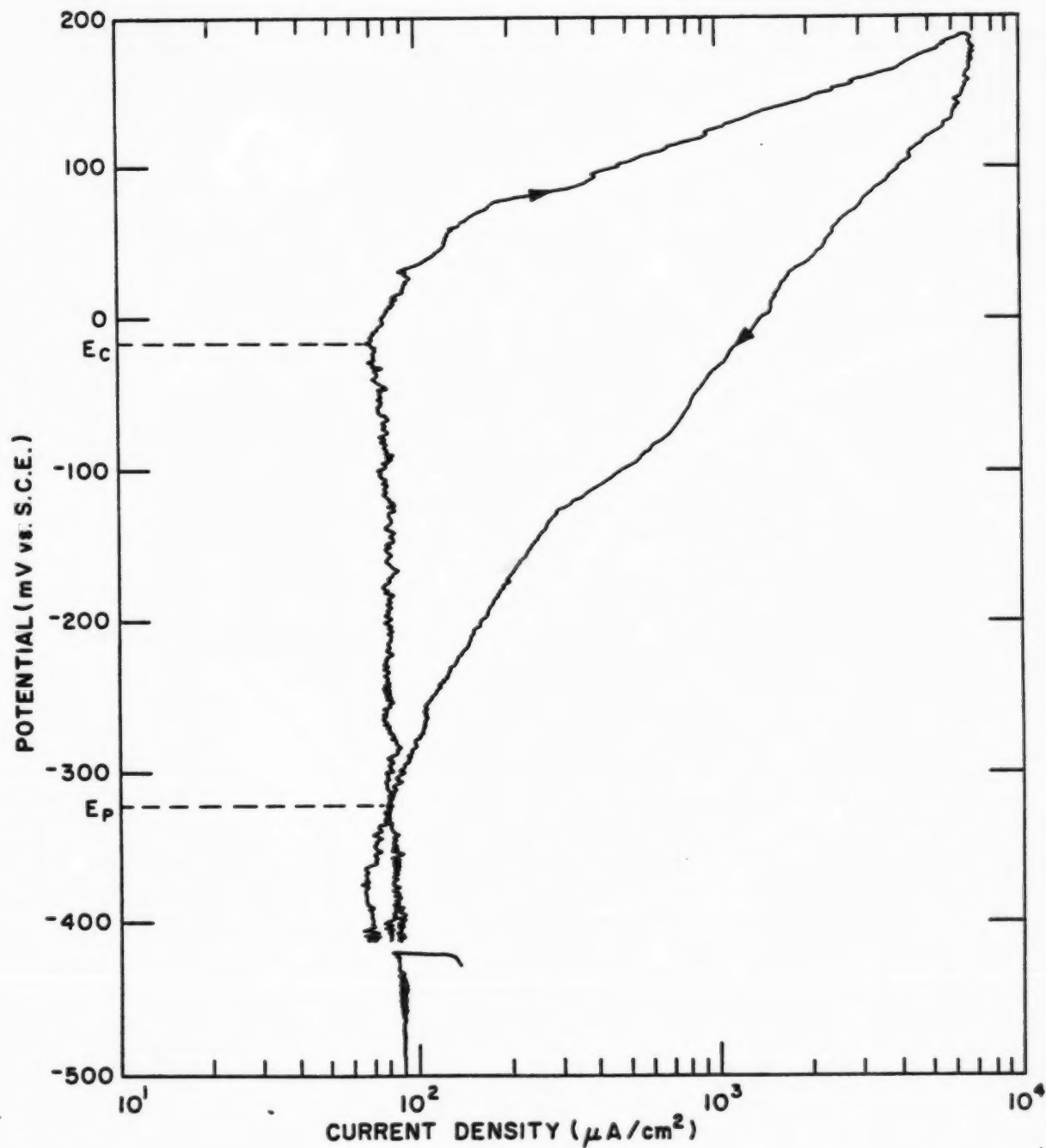


Figure 17. Cyclic Potentiodynamic Polarization curve for RS410, melt spun on a stainless steel wheel.

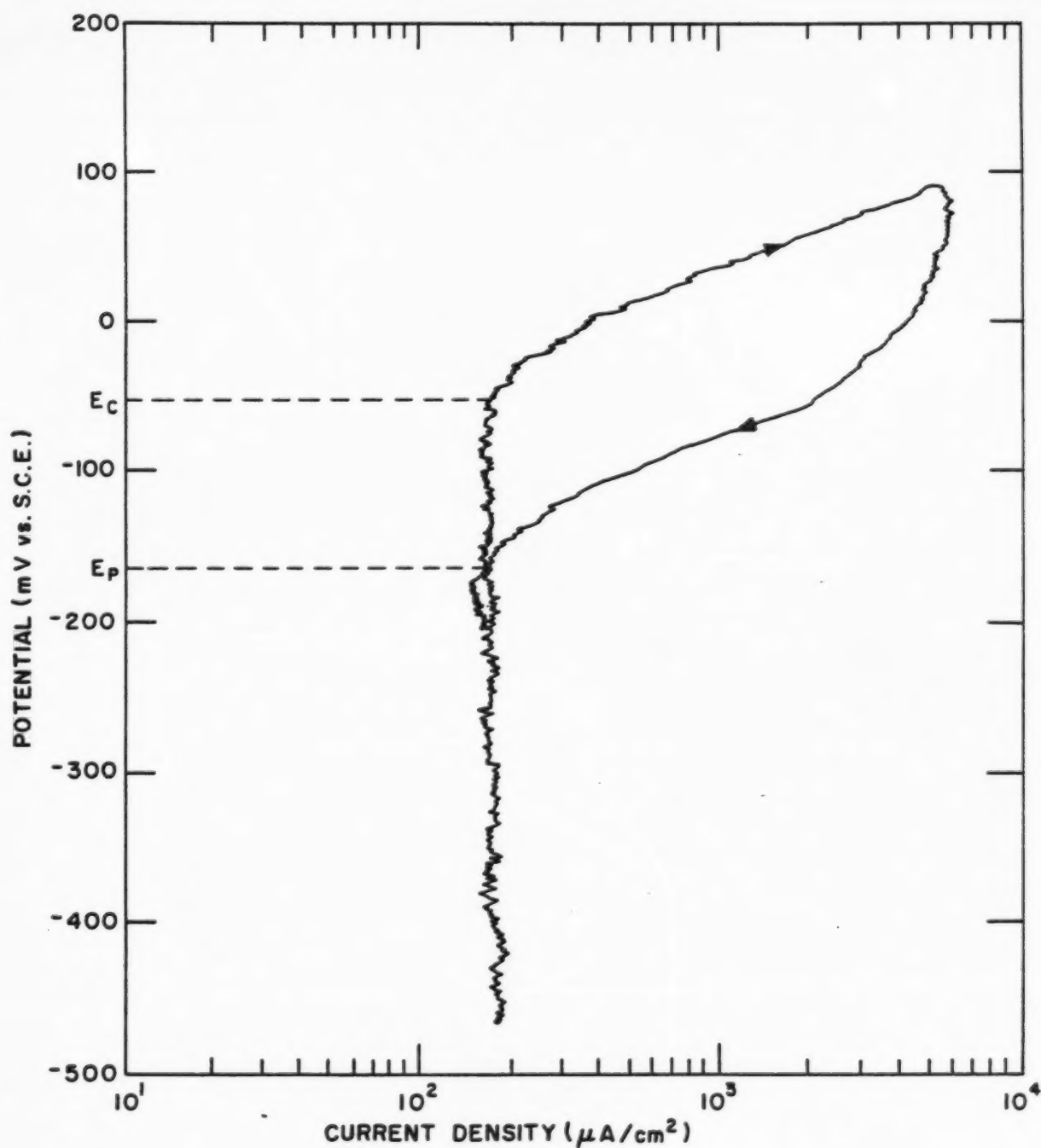
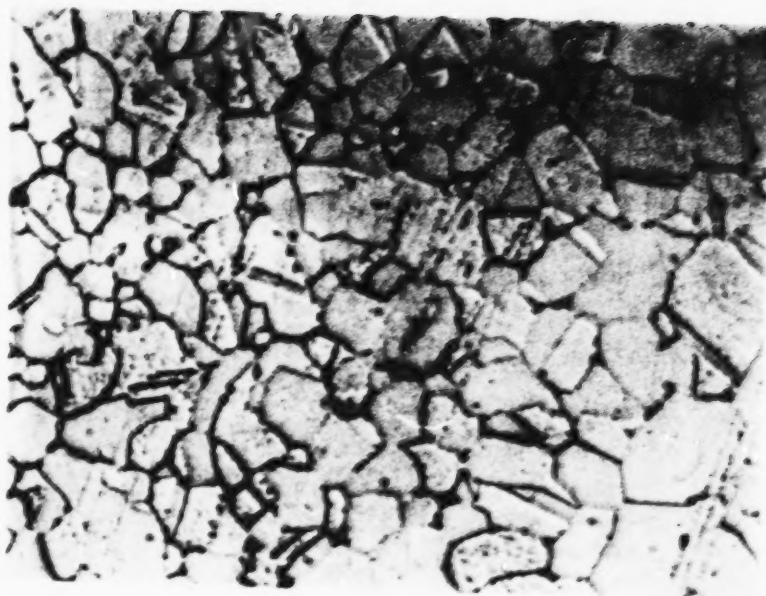


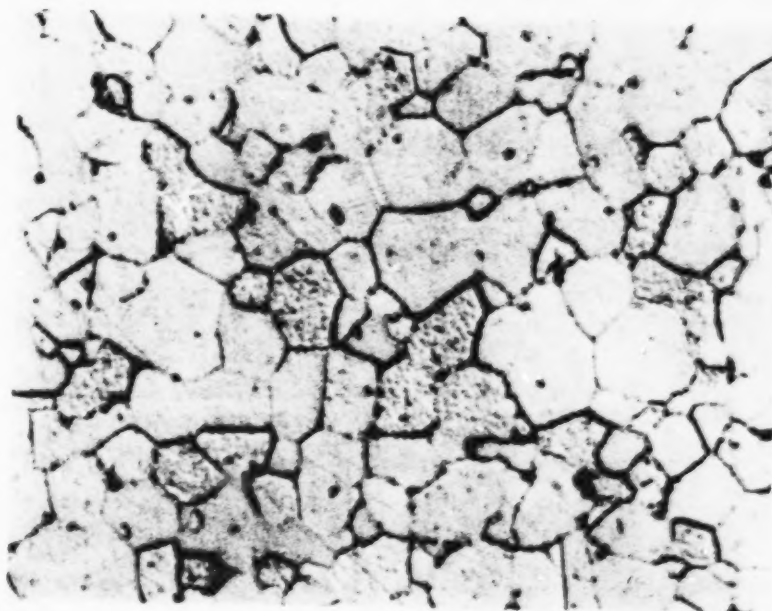
Figure 18. Cyclic Potentiodynamic Polarization curve for the low chromium content alloy, melt spun on a steel wheel.

X1000



(a)

X1000



(b)

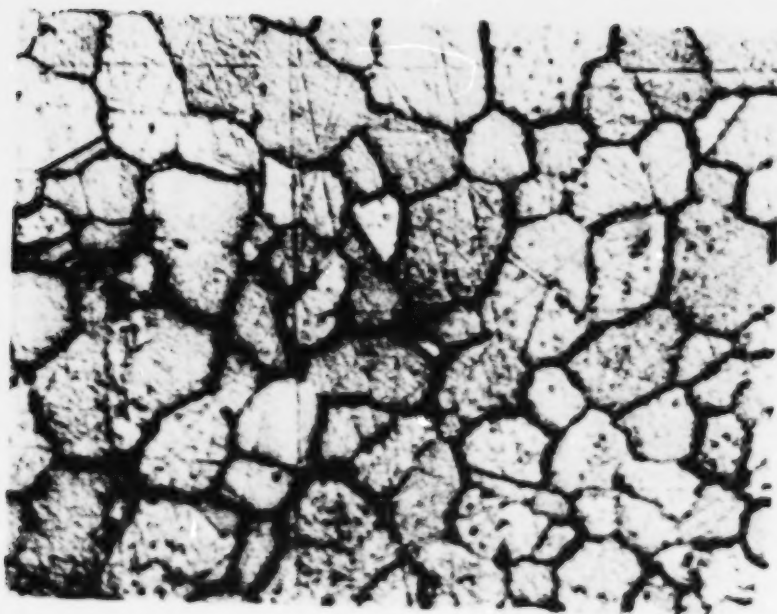
Figure 13. Microstructures of RS 304, melt spun on a steel wheel, after annealing. Annealing temperatures are: a) 600 C, b) 800 C, c) 1000 C, and d) 1200 C.

X1000



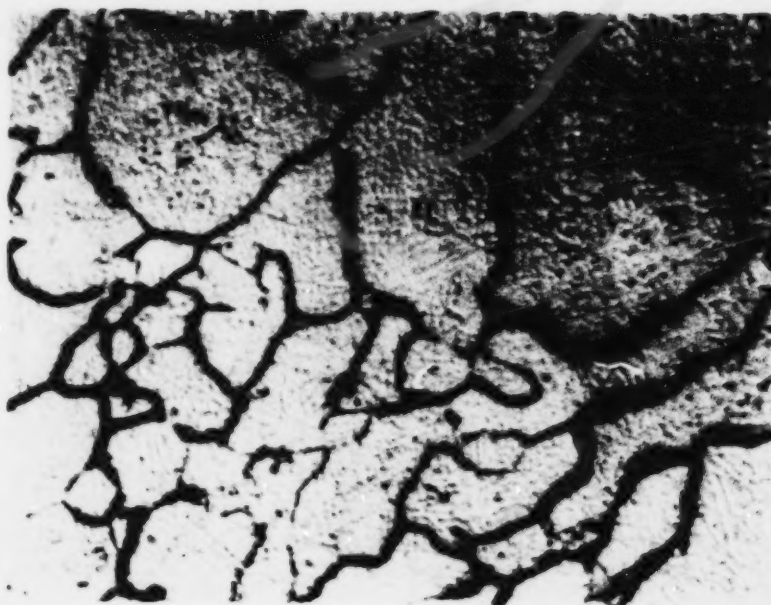
19(c)

X1000



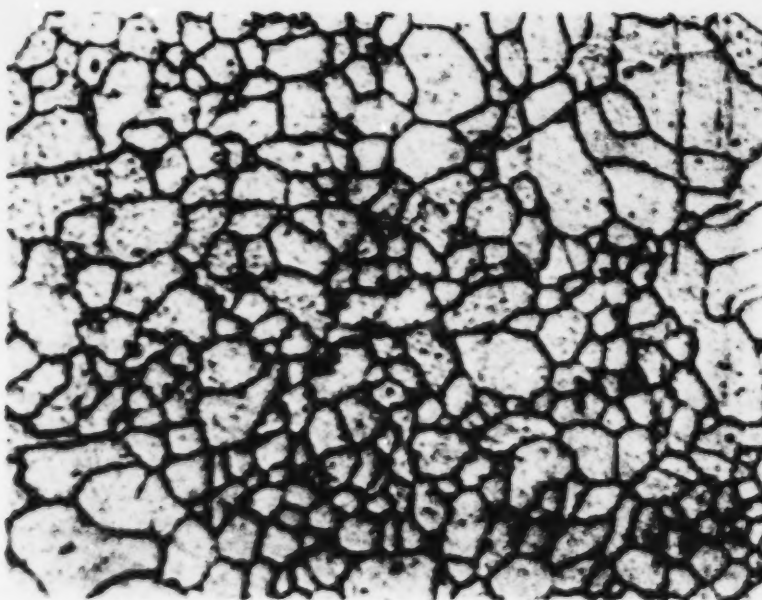
15(d)

X1000



(a)

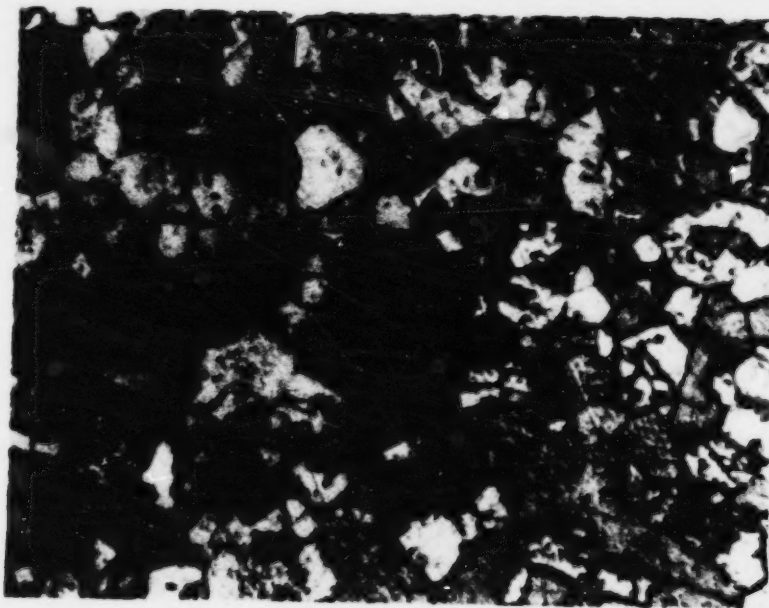
X1000



(b)

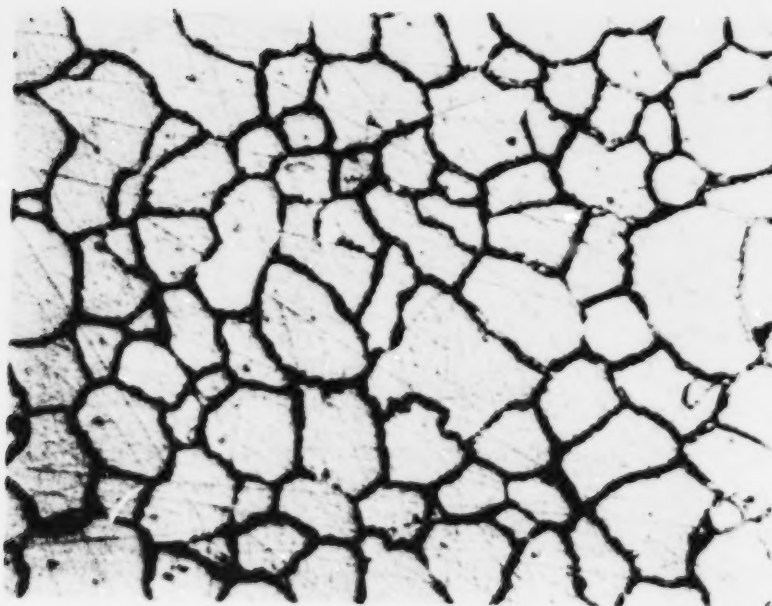
Figure 20. Microstructures of RS 304, melt spun on a stainless steel wheel, after annealing. Annealing Temperatures are: a) 600C, b) 800°C, c) 1000°C, and d) 1200°C.

X1000



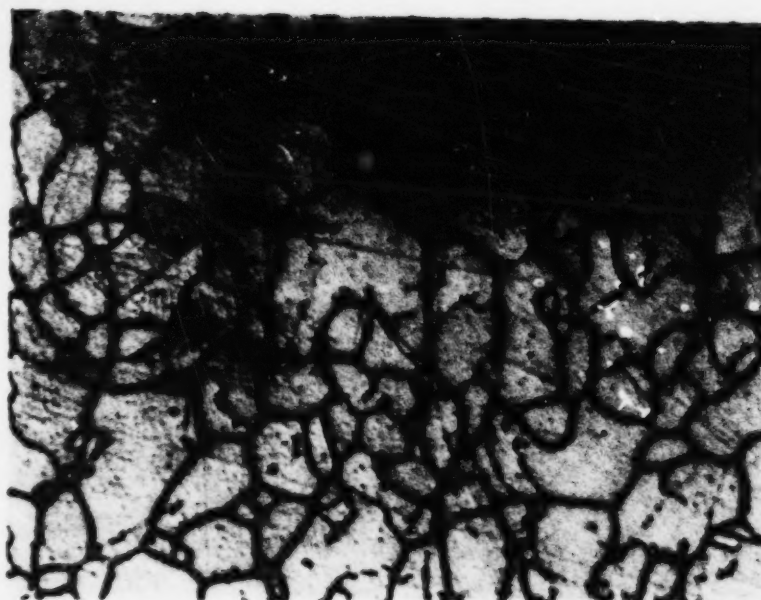
20(c)

X1000



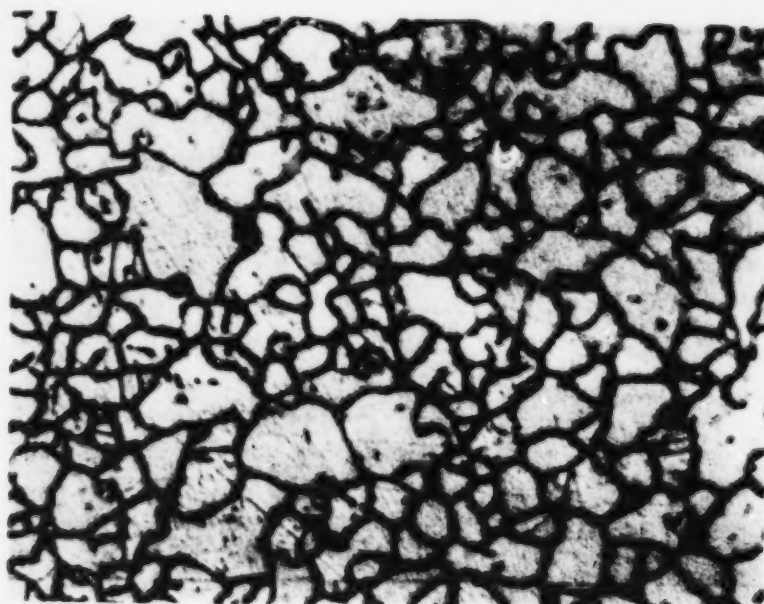
20(d)

x1000



(d)

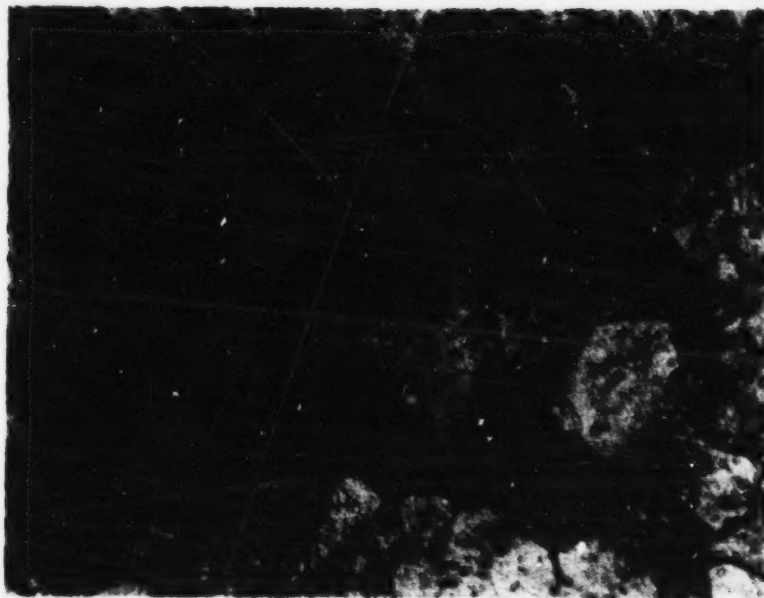
x1000



(b)

Figure 21. Microstructures of RS 316, melt spun on a steel wheel, after annealing. Annealing temperatures are: a) 600 C, b) 800 C, c) 1000 C, and d) 1200 C.

X1000



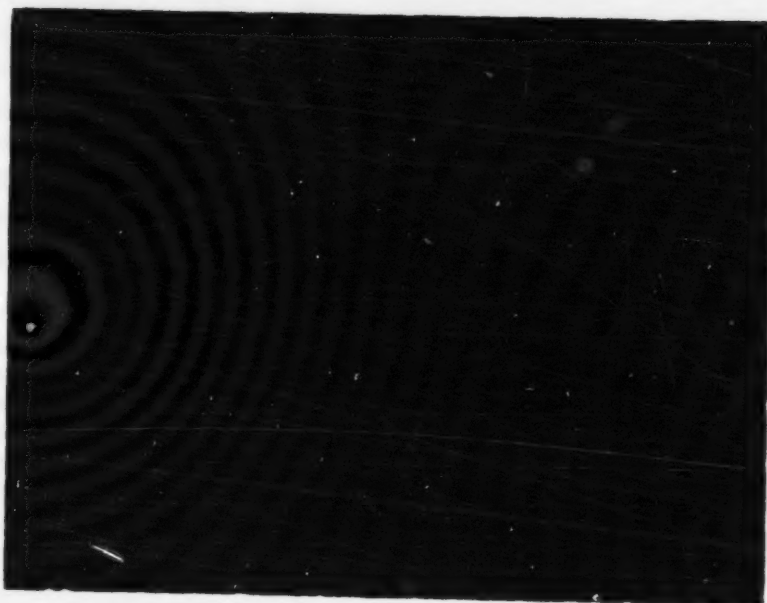
21(c)

X1000



21(d)

x100



(a)

x1000



(b)

Figure 22. Microstructures of RS 410, melt spun on a steel wheel, after annealing. Annealing temperatures are: a) 600 C, b) 800 C, c) 1000 C, and d) 1200 C.

X1000



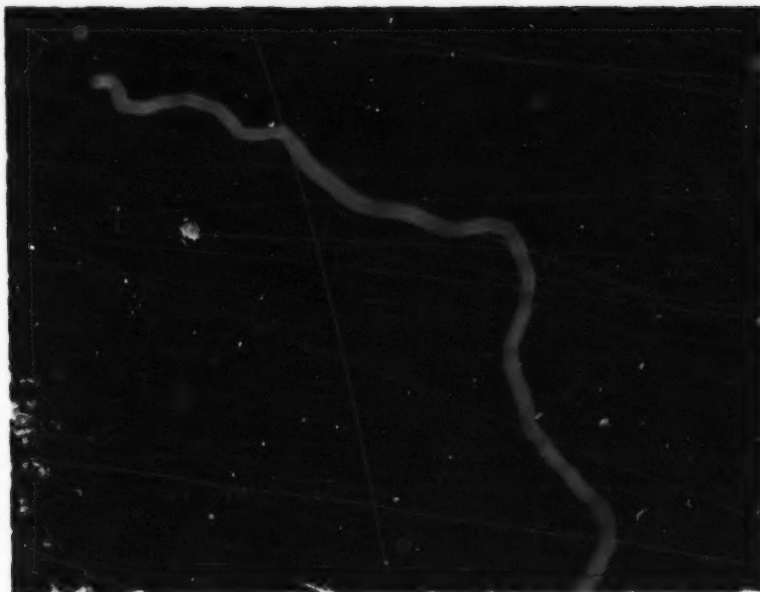
22(c)

X1000



22(d)

X1000



(d)

X1000



(b)

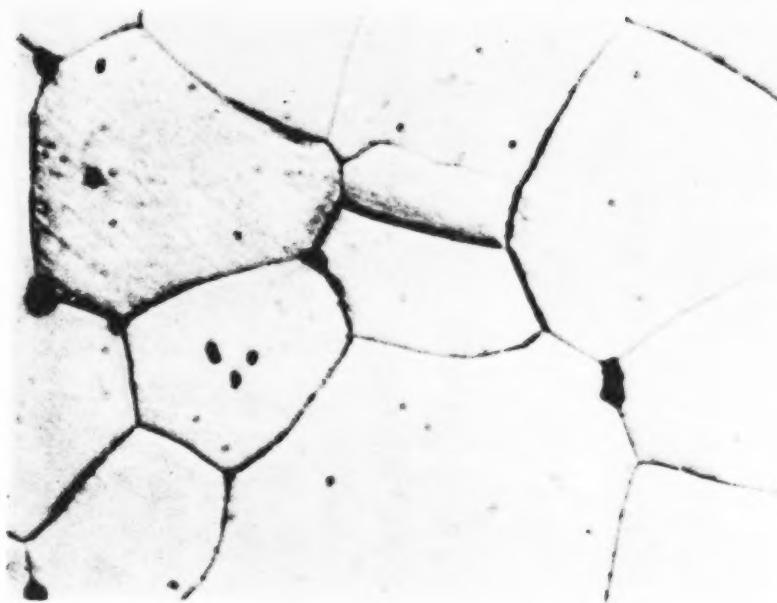
Figure 23. Microstructures of RS 410, melt spun on a stainless steel wheel, after annealing. Annealing Temperatures are: a) 600 C, b) 800 C, c) 1000 C, and d) 1200 C.

X1000



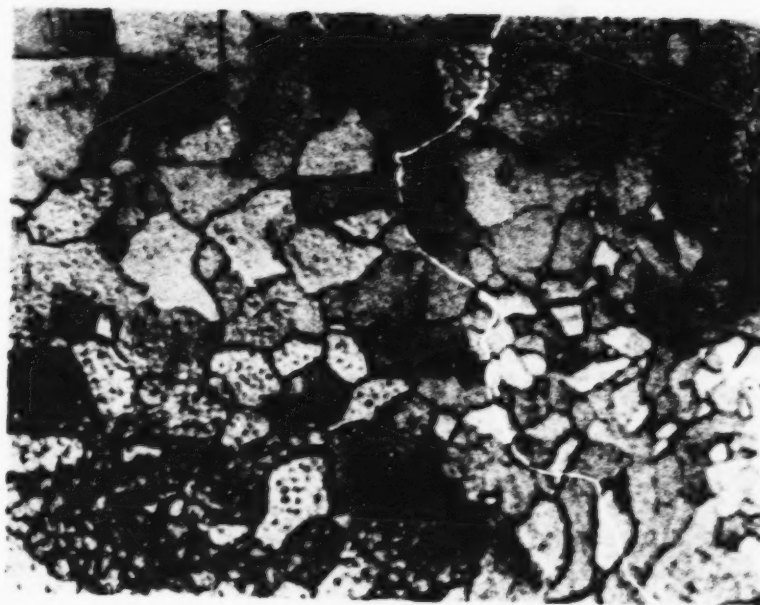
23(c)

X1000



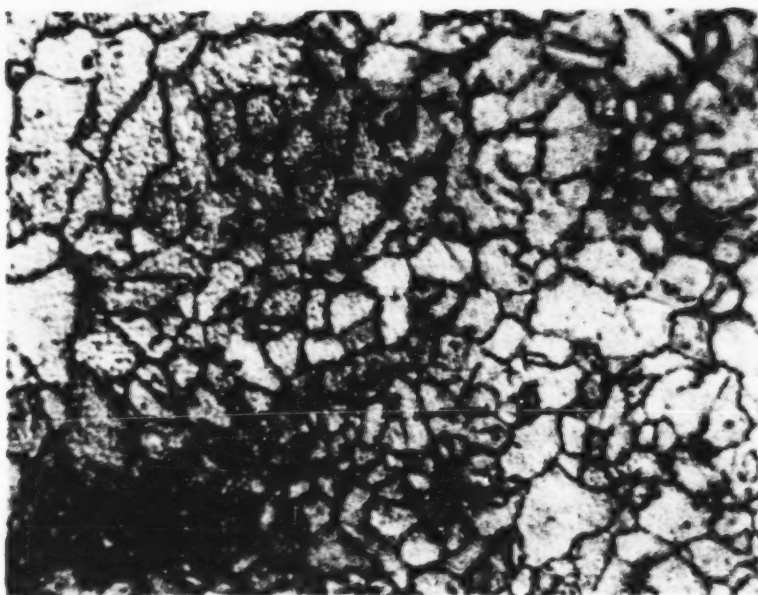
23(d)

X1000



(a)

X1000



(b)

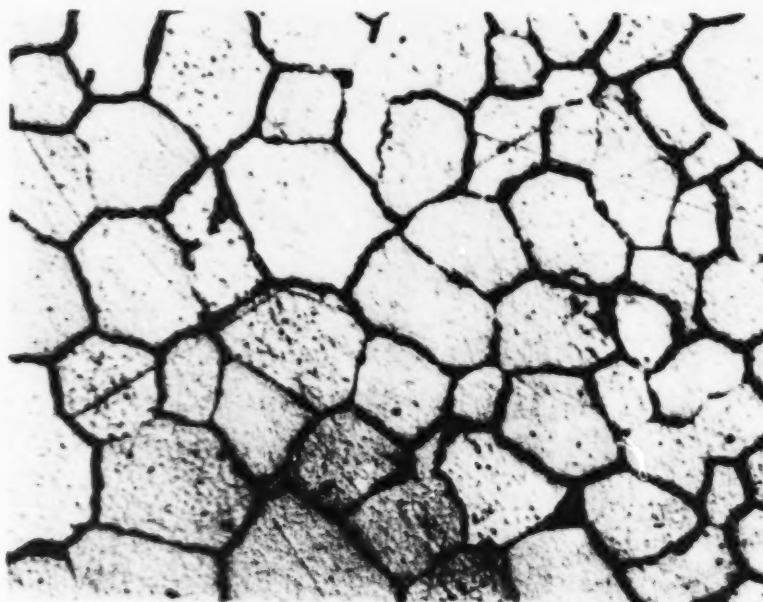
Figure 24. Microstructures of RS low chromium alloy, melt spun on a steel wheel, after annealing. Annealing temperatures are: a) 600 C, B) 800 C, c) 1000 C, and d) 1200 C.

X1000



24(c)

X1000



24(d)

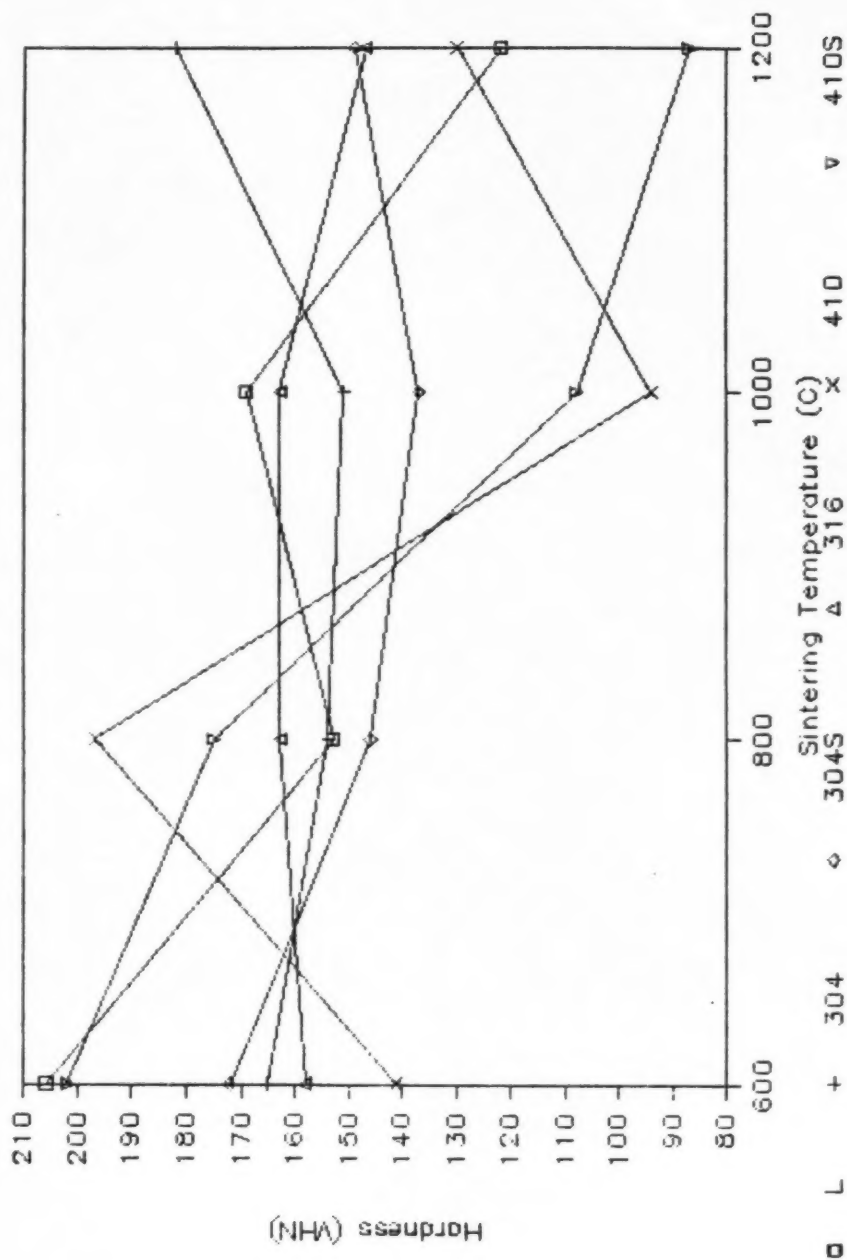


Figure 25: Microhardness measurement on annealed strips. "L" denotes low chromium. The suffix "S" indicates that the strip was melt-spun on a stainless steel wheel.

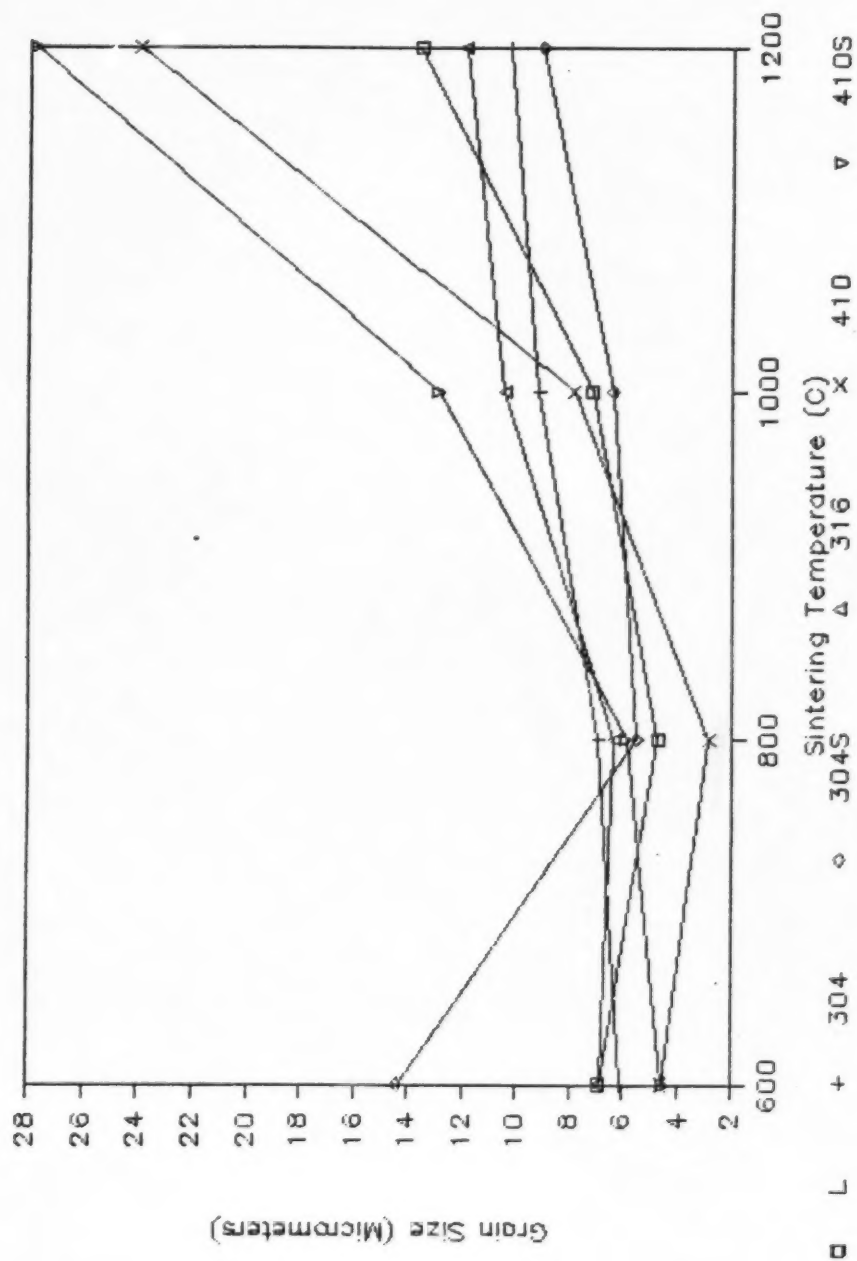


Figure 26: Grain-size measurement on annealed strips. "L" denotes low chromium. The suffix "S" indicates that the strip was melt-spun on a stainless steel wheel.

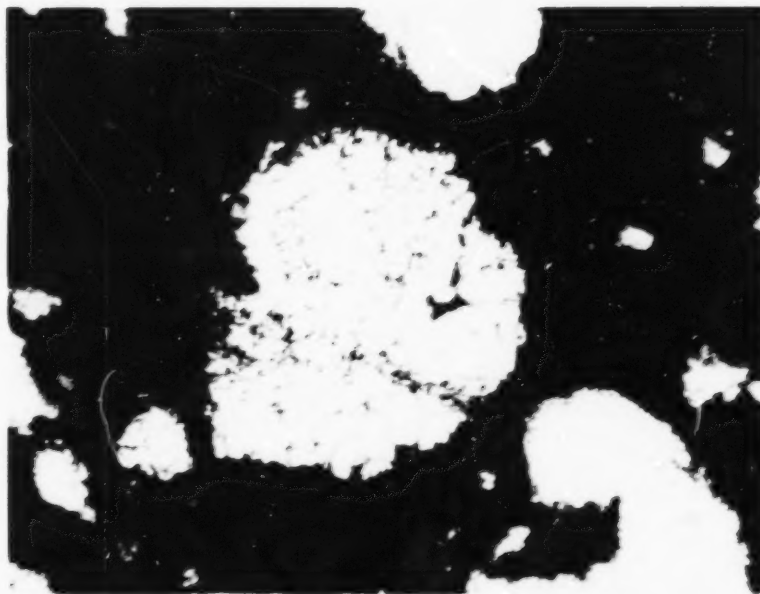


Figure 27. Microstructure of powder obtained from Metallurgical Industries (X1000).

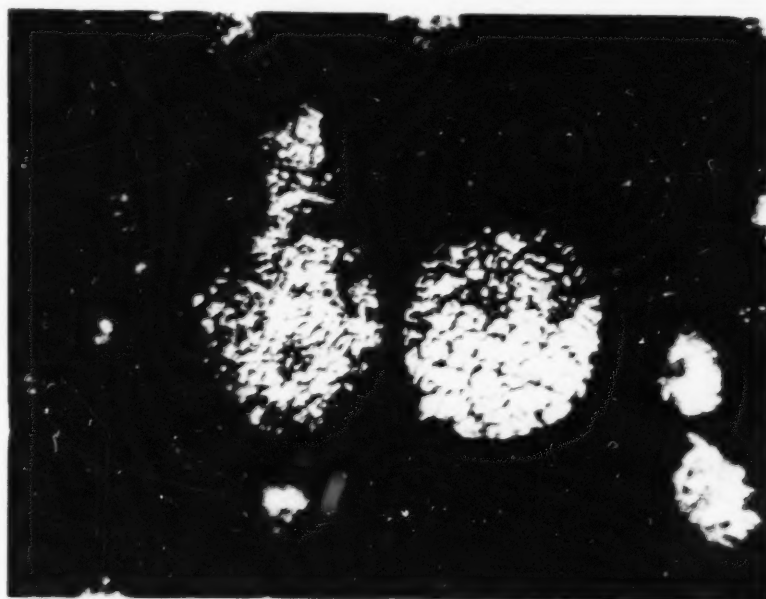


Figure 28. Microstructure of powder obtained from Pfizer Inc. (X1000).

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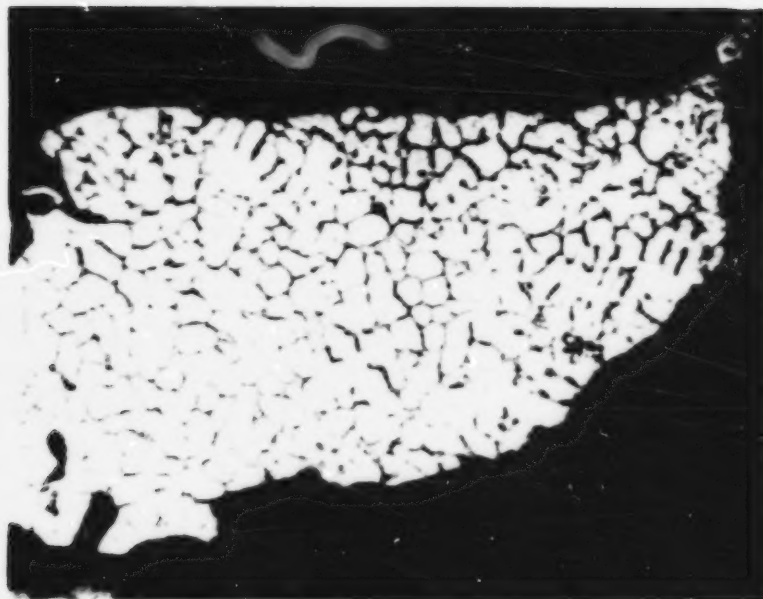


Figure 2 Microstructure of sintered material (BR-10365)